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FILTER BLANK CONTAMINATION IN ASBESTOS ABATEMENT MONITORING PROCEDURES: PROCEEDINGS OF A PEER REVIEW WORKSHOP

Workshop held April 24-25, 1986
A.W. Breidenbach Environmental Research Center
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16. ABSTRACT

Because the blank filters used to collect airborne asbestos fibers in buildings and ambient air showed a high degree of asbestos contamination, a "peer review" meeting was held to document causes and potential solutions to reduce or eliminate contamination of blank filters. The "Peer Review of Asbestos" produced a workshop type document to assist analytical laboratories and field samplers in eliminating the filter blank contamination problem. Numerous positive suggestions made in the proceedings will enhance the quality assurance and quality control aspects of sample handling and analysis.

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FOREWORD

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. The Clean Water Act, the Safe Drinking Water Act, and the Toxics Substances Control Act are three of the major congressional laws that provide the framework for restoring and maintaining the integrity of our Nation's water, for preserving and enhancing the water we drink, and for protecting the environment from toxic substances. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Water Engineering Laboratory is that component of EPA's research and development program concerned with preventing, treating, and managing municipal and industrial wastewater discharges; with establishing practices to control and remove contaminants from drinking water and to prevent its deterioration during storage and distribution; and with assessing the nature and controllability of releases of toxic substances to the air, water, and land from manufacturing processes and subsequent product use. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

Because the blank filters used to collect airborne asbestos fibers in buildings and ambient air showed a high degree of asbestos contamination, a "peer review" meeting was held to document causes and potential solutions to reduce or eliminate contamination of blank filters. This workshop-type document will assist analytical laboratories and field samplers in eliminating the filter blank contamination problem.

Francis T. Mayo, Director
Water Engineering Research Laboratory

PREFACE

In April, 1986, the USEPA's Office of Research and Development, Water Engineering Research Laboratory (WERL) hosted a peer review of procedures used in WERL's monitoring of an asbestos abatement activity in which blank contamination problems were observed during use of the EPA-approved Yamate Method for Transmission Electron Microscopy on Nuclepore Filters.

The peer panel represented a cross-section of national expertise in electron microscopy and asbestos analyses.

We were quite pleased with the reviews. The Panel worked very well together and in a constructive tone addressed the problems encountered.

Tom Powers has solicited and collated copies of the presentations made at the peer review and has included the summary comments and recommendations of the peer panel heads, Steven Hayward (State of California Air and Industrial Hygiene Laboratories) and Eric Steel (National Bureau of Standards).

Because we believe that these issues, problems, and recommendations are of sufficient interest to the user community, we are distributing these proceedings on a limited basis.

For additional information, please contact Tom Powers (513-569-7550) or me (513-569-7509).

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PEER REVIEW OF THE HIGH BLANKS ASSOCIATED WITH AN ABATEMENT STUDY

Reviewers:

Steven Hayward
Air Industrial Hygiene Laboratories
State of California

and

Eric Steel
National Bureau of Standards

General Purpose

The purpose of the peer review is to consider the possible causes and solutions to the apparently high concentrations of asbestos found on field blanks during a recent study of asbestos abatement procedures. A meeting was held on April 24 and 25 1986 to discuss the data and general problems associated with TEM analysis of asbestos samples. Data presented to the peer reviewers can be found in Appendix I.

We have divided the text into two general sections, one dealing directly with blank problems and the other dealing with the comments on general TEM analysis of asbestos samples. As will be shown, these two categories both need to be clarified to help prevent similar problems from occuring in the future.

SECTION I

PROBLEMS OF APPARENT HIGH BLANK LEVELS IN ASBESTOS ANALYSIS OF AIR SAMPLES

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Definitions:

Several types of blanks were discussed at the meeting and in order to keep the terminology consistent and clear the blank names and short definitions are given below.

Blank: Any filter which has not had air drawn through it to sample for asbestos.

Laboratory Blank: A filter analyzed directly from the manufacturer's filter box. This blank looks at contamination due to filter and electron microscope sample preparation only.

Field Blank: A blank taken into the field with the actual sample filters but does not have air drawn through it. These blanks are contained in sampling cassettes and can be used two ways. The cassettes can be either closed or open throughout the sampling period. Closed field blanks are controls for contamination during the filter-cassette assembly, cassette handling in field and laboratory. Open cassettes also include any contamination during sampling that is not due to air being drawn through the filter, e.g. contamination through static charge or dust fallout onto the filter during handling or air sampling. These two types of field blanks will be referred to as closed-field blanks and open-field blanks respectively.

Summary of Results

A brief summary of the study as it is understood by the reviewers is presented here and is used as a basis for the conclusions and recommendations listed below.

The blanks discussed at the meeting were closed-field blanks prepared at the P.E.I. office building. It was reported that this building is relatively new and contains no apparent asbestos building material. The blank cassettes were prepared along with air sample cassettes by various personnel at various times throughout the study. No clean-room or clean-bench conditions were observed. No analysis was made of the batch of filters prior to the filter-cassette manufacture at the office building.

The initial closed-field blanks labelled sample group 1 were analyzed by McCrone and IITRI and were found to have approximately typical Nuclepore blank values on the order of 2 asbestos fibers per 10 grid squares (F/10GS). Most subsequent analyses by the McCrone laboratory showed relatively high field blank data with a typical analysis being 20 asbestos fibers per 10 grid squares. During the same time period James Millette of McCrone told us that internal laboratory blanks (from a different batch of Nuclepore filters) were the more typical loading averaging less than approximately two F/10GS.

Other laboratories have analyzed some of the field blanks from the same study. ETC in seven analyses found an average of 5 F/10GS but this slightly high value was due to one high filter having 25 F/10GS. This appears to be contradictory to the relatively constant high asbestos values found by McCrone, but since no filters were analyzed by both laboratories no direct comparison is available.

NIOSH and EPA results of some of the same filters analyzed by McCrone show that there is a major discrepency in either the format for reporting results or the actual results. Both NIOSH and EPA report much lower counts of definite asbestos fibers with much higher "nonasbestos" and "NOID" fibers than those listed by McCrone. However, after talking to the analysts at NIOSH and EPA, it is felt that the identification results may be biased by lack of experience in asbestos analysis.

This data was supplemented by reports from Richard Lee of ETC stating that his laboratory has found consistently low values for Nuclepore laboratory blanks. James Millette verified this data adding, however, that in two cases he has found elevated blanks in specific boxes of Nuclepore filters. In all cases these labs find short, thin asbestos fibers to be typical. George Yamate of IITRI said that laboratory blanks for Nuclepore filters appears to be increasing over the years and that recent batches often contain relatively high levels of asbestos. Yamate also showed size distributions for his blanks that included relatively long fibers and bundles.

Blank study conclusions:

We feel the results of the blank study are inconclusive. There are two possible causes for the reported high blanks, 1) the filters were contaminated with asbestos; and/or 2) the identification of asbestos was incorrect at McCrone.

With the exception of the first set of filters analyzed by McCrone and IITRI, we have no reliable consistent replicate laboratory data with which to determine the validity of the McCrone analysis. There are large discrepencies between reported asbestos values from McCrone, ETC, NIOSH, and EPA, but with the data presented it is impossible to determine the cause of the discrepencies. There are several reasons for this:

1. the data was not reported in a consistent format

2. the fibers were not analyzed in a consistent way

or 3. one lab (ETC) did not analyze the same filters as McCrone.

Without first validating the McCrone analysis no conclusion can be drawn as to the overall asbestos contamination level of the closed-field blanks or the contamination source. The quality assurance steps taken by McCrone, such as the low laboratory blanks during this time period, imply that there are probably more fibers on the filters, but the discrepency with other laboratories reporting makes it impossible to draw any further conclusion as to the type of fibers. If the McCrone results are correct then the high levels could be from the original filters or some step in the preparation and use of the filters. If the McCrone results are incorrect then steps need to be taken to correct the method of identifying fibers and the filters would need to be reanalyzed.

If the McCrone results are valid, then asbestos contamination of the filters occured. The major possible sources of contamination are:

1. Nuclepore filters 2. Millipore cassettes 3. P.E.I. office or personnel contamination during cassette preparation. 4. Sampling site contamination of the outside of the cassettes 5. Sample mislabelling(with real air samples) or mishandling(opened in contaminated areas) 6. contamination during transportation,shipping, packaging, receiving 7. contamination during sample preparation from the outside of the cassettes or from the McCrone personnel or laboratory.

Recommendations for the Blank Study

There are two unresolved questions: 1) is the McCrone identification and counting data correct? and if correct then 2)where does the contamination come from?

In order to answer these questions in an efficient and useful manner, we feel several TEM grids should be prepared from one of the high blank samples such as sample group 3,4,9, or 10. These grids should then undergo analysis by several well trained analysts in several laboratories. All these laboratories should use the same identification protocol and take electron micrographs to demonstrate the validity of the analysis. The grids should be prepared by an outside independent laboratory. A suggested protocol can be prepared by the Bureau of Standards if requested. This interlaboratory analysis should answer the question whether the McCrone analysis is correct.

If the McCrone analysis is correct, then the results of the P.E.I. study are questionable. This is based on the information presented by Ann Crone stating that the high field blank levels overlapped with the results from the air samples. Further study could clarify whether the asbestos contamination was from the original filters or some other source, but would probably not be able to correct the results in the study. We feel it may be more beneficial to correct the the general laboratory and sampling procedures for future studies than it is to try to find the contamination source in this study. The next section lists some general recommendations which would help alleviate these problems and make contamination tracing possible in future studies.

However, if there is a specific interest in determining the source of the contamination in this study, then several steps may be taken. The first is to analyze the filters left in the box to determine if these have the high asbestos contents. If these filters have high asbestos concentrations then they were either contaminated in the manufacturing process or in the handling at the P.E.I. office building. Further work looking at the size distributions and types of fibers can be performed, as urged by Richard Lee. These distributions can be compared with the site's air sample filter and bulk sample data to try to determine if the sources are similar. This analysis is difficult because, if the blanks are valid blanks, then the air filters have both the air and contamination fibers on them. It is not clear to the reviewer's that useful information will come from this research.

It is felt that not enough data exists on the location and handling of the blanks to identify a unique contamination location or source.

The last recommendation regarding the specific study is to use the TEM to analyze the Millipore filters from the PCM monitoring using the method of Burdette and Rood. Analyzing the blanks first and cleaning the cassettes off before entering the clean room should help to alleviate some of the problems observed in this study.

General Recommendations for Blank Laboratory Procedures.

The first requirement for any study of asbestos in air should be to verify that the materials and methods used will bias the results as little as possible. To this end, the filters used to collect the air samples should be analyzed and certified "clean" of asbestos or more reasonably to have a defined quantity of asbestos that is low enough that it does not interfere with the analysis. The asbestos levels found on these blank are one of the most important parameters in defining the best level of detection attainable. We feel the most efficient way to attain characterized and low concentration blank filters is to work with the manufacturer to certify a filter lot by preparing the filters in as clean a manner as possible and then characterizing a statistical number of filters and fibers to get a certified concentration and uncertainty of asbestos. If possible this characterization would also include the mounting cassette. These "certified" filters in cassettes would then be set aside for asbestos sampling only and either sold or given to laboratories carrying out asbestos analysis projects. EPA has done this in the past with other types of filters in other analytical areas. The increased cost of these filters would have a minimal effect on the overall cost of the asbestos analysis.

Several other less desirable possibilities for providing characterized filters exists. Currently most of the labs present at the meeting prefer to analyze the batch of filters previous to their use. However, it appeared that most of the labs analyzed 1 or 2 filters to characterize the filter lot. The statistical uncertainty of the results of this analysis is not well defined. Should this practice be continued it is suggested that a protocol be written to attain a defined and consistent acceptable concentration and uncertainty of asbestos on the blank filters before air sampling is begun. This concentration used with sampling and analysis data could be used to define a limit of detection for asbestos in the specific project. In comparison with the first choice of a "certified" filter lot this involves redundance of analysis at different laboratories and increased expense in asbestos analysis.

Another possibility exists in "cleaning" the filters prior to use. This would include washing and agitation with water or other more caustic agents. The filters at this time would again have to be characterized for asbestos. The solutions would have to be characterized as asbestos free and the solutions would become contaminated by the filters so that the cleanliness of the filters may be a function of time of washing or number of filters washed. While research in the method would be useful, especially if the detection limit of asbestos in air is to be lowered, there is currently insufficient data to recommend filter washing. This may be a useful approach for the "certified" filter lot.

Once a characterized and low asbestos concentration filter lot or box exists further clarification and standardization is needed in the handling of the filters. This includes defined clean room and/or clean bench procedures. If possible the "certified" filters could be premounted in conductive sampling cassettes and the characterization of blank asbestos levels would include this step. It is felt that definite procedures should be available to laboratories that prepare the sampling cassettes or analyze the filters. This would include requirements and specifications for a clean room or clean bench as well as methods of monitoring the clean area. Other parameters that need to be clarified include the clean area entrance procedures for both people and objects. For instance, people working with bulk asbestos samples or coming from areas with bulk asbestos samples may contaminate the clean area, also filter cassettes coming from asbestos sampling sites may contaminate the area. Therefore before entry into the clean area, the people and objects may need to be decontaminated.

Until or if a "certified" filter lot can be attained the following rules should help to reduce the types of problems seen in this study.

1. Identify and create a clean area for filter handling in an area that is well defined and completely separate from areas and personnel that handle bulk asbestos.

2. Routinely monitor this clean area with air filters and fallout samplers as well as the more typical clean room particle counters. (If contamination is seen, find and eliminate its source.)

3. If possible, buy the cassette and filters premounted. (Ask that the filter lot number be specified.)

4. Characterize the filters for asbestos from the box or lot or cassette assembly prior to an air sample study.

5. If cassettes and filters are put together by the laboratory rather than the filter manufacturer, then the cassettes should also be checked for contamination.

6. If the the filters average 5 or more asbestos fibers per ten grid openings return the lot to the manufacturer.

7. Keep a written log or record of all blank values and filter lot numbers and clean area monitor information.

8. All personnel entering the clean area should wear clean, non-asbestos contaminated clothing. A common clean room practice is to put on clean outer garments, such as a lab coat and boots, before entering the clean room. Limit access to the clean area to as few personnel as possible and personnel that are aware of the contamination possibilities. personnel that have handled bulk asbestos or have been to contaminated sites should not be allowed in the clean area.

9. Decontaminate all objects brought into the clean area. For example, do not bring the samples into the clean room in a box that was used to carry the samples from the sampling site to the laboratory. This may also mean using a HEPA vacuum, latex stripping, or washing the filter cassettes before entering the clean area.

10. Verify all sample preparation materials are asbestos free or at least non-contaminating. This includes the Jaffe wick materials, grids, etc.

11. Routinely analyze laboratory blanks on a set of filters that have previously been characterized. This monitors sample preparation contamination.

12. Use consistent reporting and identification format (see general asbestos analysis recommendations.)

13. Field blanks and samples should have a record of every location and person associated with storage, usage, or transportation. There should be a field blank for every location. As an example in the P.E.I. study, there should be blanks uniquely associated with the P.E.I. office building, transportation to the sampling site, storage at the site, as well as the specific air sample blanks. In this way the contamination sources can be traced and corrected. These blanks should be handled in the same manner as the samples in that area. For instance, closed field blanks for storage areas, and open field blanks for air sample areas.

14. Again, people handling bulk asbestos containing materials in the field should not handle the sampling devices or blanks.

15. Field blanks should be analyzed first and early in the sampling program to identify and eliminate contamination problems as early as possible. Note that not all the field blanks need to be analyzed. A logical and economical approach would be to analyze the first field air sample blank as early as is technically feasible in the project. Then, if this blank shows elevated levels the other location blanks can be used to track down the contamination. However, if the first blank is near the certified value then the other blank values can be presumed to be low.

SECTION II

GENERAL ISSUES OF SAMPLING AND ANALYSIS

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Introduction

During the course of the peer review of the field blank contamination problem, several general points came to light regarding deficiences in current sampling and analytical practices. Although the peer review was not specifically convened to critique current methods, these deficiencies were so glaring that we as reviewers felt compelled to note them. Further, we strongly urge that these suggestions be incorporated in some way into any new TEM method written by any government agency and/or voluntary standards organization. This might include the methods currently under development by the ASTM and by NIOSH. It is further urged that any new method be published in loose-leaf format, to allow for the possibility of upgrading portions of the method as new scientific or technical information becomes available. This is clearly preferable to the U.S. EPA's approach of commissioning an entirely new method every time a revision is required.

Following are our comments on each of these general issues of sampling and TEM analysis for airborne asbestos:

1. Comparison to Phase Contrast Microscopy Disclaimed

Very often people who have no technical background equate asbestos concentrations determined by TEM with numbers obtained by phase contrast microscopy (PCM). Unfortunately this misunderstanding has also been demonstrated by people with some technical expertise. For this reason, we urge that any TEM methodology for quantitating airborne asbestos contain a disclaimer in the beginning. This disclaimer should be in boldface, and should state that airborne asbestos concentrations determined by the method are not equivalent to concentrations determined by PCM.

2. Sampling Protocol

Any environmental analytical method is complete only when it is seen in the perspective of sampling and analysis. Enough is known about sampling for airborne asbestos that it seems sensible to standardize the sampling protocol, and include it in the method. This will prevent variations in sampling methods from affecting the results of analysis. It would also allow the method to specify in detail any procedures for contamination control, or any other aspects of sampling necessary for quality control/quality assurance. Following are some of the more important aspects of sampling that should be included in a standardized protocol:

A. Sampling Details

It is unlikely that fine details of sampling, such as the brand of sampler, should be standardized in the method. However,such things as cassette type, cassette orientation during sampling, range of acceptable flow rates, and acceptable methods for flowrate measurement or calibration should be specified. It is also crucial that the sampling and analytical community resolve

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the issue of filter type. Lately there has been a revival of interest in the use of tortuous pore filters, as reported by Burdett and Rood. The advantages of use of these filters are many, including compatability between all three types of microscopy (LOM, SEM, and TEM), transport without loss, less charging during sampling, transport, and handling in the laboratory, easier handling, and possibly less contamination (according to Rich Lee's experience). A current version of this method should be tested against the Nuclepore method, and a standard method should be settled upon once and for all.

B. Charging/Grounding

At the Peer Review session, Paul Baron showed strong evidence that charging of insulating cassettes can produce erroneous results which are likely to depend upon flow rate, relative humidity, and several other variables. Our laboratory (in the CA Dept. of Health Services) and several other laboratories have found fibers adhering to plastic cassettes, presumably because of the static charge developed on the cassettes during sampling and/or transport. Data presented by Dr. Baron suggest that all sampling should be carried out with conductive cassette inlets or cowls, and that these should be grounded whenever possible. Any sampling protocol should therefore include the use of such measures.

C. Contamination Control

All details of filter handling, both before and after sampling, should be spelled out in detail in the protocol. This is so that contamination can be controlled, and so that, if some of the blanks are found to be contaminated, the source of the contamination can be found. Some of the steps in contamination control would include 1) details of laboratory blank preparation, 2) cassette cleaning and loading procedures, 3) cassette handling in the field, 4) sample transport 5) sample and personnel decontamination for entry into the laboratory clean preparation area, 6) laboratory clean area work practices, including filtration of reagents, cleaning glassware and implements, contamination control during carbon coating and filter clearing, sample storage, etc.

3. Analysis

Discussion during the Peer Review meeting brought up many aspects of analysis that have not been well standardized, and that are overdue to be included in an updated method. The most notable of these are listed below:

A. Length Lower Limit

Work by Small and Steel has shown that interoperator variability can be decreased substantially if extremely short fibers are not counted. It therefore appears prudent to have the method specify that only concentrations of fibers greater than some length lower limit should be reported. This lower limit might be arbitrarily set to 0.5 micrometers. In fact, a 0.5 micrometer long chrysotile fibril (the thinnest fiber possible) is likely to have an aspect ratio slightly higher than 10.1, which is a number that some have suggested as a lower limit for calling something a fiber. Although there may be some who claim that fibers shorter than 0.5 micrometers actually pose some health risk, attempting to quantitate them makes no sense if doing so increases the relative size of the confidence interval (the coefficient of variation) precipitously. The method could, of course, allow for recording the detection of such "fibers" if it is so desired.

B. Magnification

Two issues about magnification came out at the meeting that should be dealt with in a standard method: a permissible magnification range, and the addition of a low magnification scan.

Permissible Magnification Range

There was some argument about whether 10,000 times is a suitable magnification for detecting all asbestos fibers. This discussion pointed out the fact that the choice of 20,000 times is somewhat arbitrary, but that few samples have been multiply analyzed at several magnifications to determine a range within which the accuracy is not compromised. Ultimately, a standard method should specify a permissible range of magnifications. It should also make a careful distinction between nominal film magnification, nominal screen magnification, and calibrated screen magnification. It is the latter number that should be expected to be within the permissible range (unless, of course, the scanning is done through the binoculars).

Low Magnification Scan

TEM can be used in a manner that bridges the gap between the NIOSH phase contrast microscopy (PCM) methods, and TEM as it is normally practiced. This can be done by scanning samples at low (400x-2000x) magnification, and recording only fibers that would normally be visible and countable by one of the standard PCM methods (length >5 micrometers, width >0.3 micrometers, and aspect ratio >3:1). The advantage of using the TEM for this type of analysis is that the entire TEM arsenal of identification tools is available, so that only asbestos fibers will be counted. Some of us feel that it is critical that a database of such counts and their relationship to PCM and total TEM counts should be built up. Once grids have been prepared, such an analysis takes only approximately as long as a PCM count because the field of view size can be similar. This count is also useful as part of an overall TEM analysis simply because it allows the large tail of the fiber size distribution to be defined with improved statistical certainty.

Counting and Identification Criteria

The method that was developed by George Yamate, intended as an updated version of the EPA Provisional Methodology, specifies unambiguously (within the framework of three levels of analysis) how fiber identification is to be carried out. It is evident that some laboratories have developed their own nomenclature for these categories. It is also clear that some operators have more expertise than others in using the analytical tools on their microscopes, and further that some microscopes have more and/or better tools available for fiber identification than others. These three problems have separate solutions: variable operator ability can only be dealt with by some kind of operator training/certification requirements (see below). Differences in laboratory identification and categorization criteria would be eliminated if all laboratories were required to (or agreed to) use one standard method. And poor analytical instrument capability can only be dealt with by requiring some minimum of instrumentation.

Beam Dose, including Microbeam Diffraction

It became clear from the descriptions of radiation damage in chrysotile fibers that most microscopists are not attempting to minimize electron flux in any way. Electron flux is easily minimized such that radiation damage, both in diffraction and imaging modes, is not detectable over many minutes of observation. Such conditions do not produce loss of contrast, since contrast is simply a function of the sample and the imaging mechanism. They also produce no reduction in visibility, as long as the operator is allowed sufficient time to become dark-adapted (approx. 20-30 minutes) at the beginning of the counting session. Low dose techniques are also critical if microbeam diffraction, rather than

SAED, is used for identification. This is because periodic exposure to a very bright image results in loss of dark adaptation. Since microbeam patterns are somewhat weak (but high contrast), this loss of dark adaptation prevents perception of the diffraction spots. The same result occurs when the operator turns on anything but a red light in the microscope room for, e.g., recording the dimensions of a counted fiber. Such aspects of low-dose technique should be included in a standard method.

As was first shown by Beaman, microbeam diffraction produces far more consistent results with chrysotile fibers than does SAED, especially in the presence of extraneous material. The CA Dept. of Health Services lab's experience is that virtually every chrysotile fiber can be made to produce an identifiable microbeam pattern, as long as a portion of its length equal to approximately 0.04 micrometers is unobscured. Most TEM's can be set up to do microbeam diffraction without much difficulty. It therefore seems reasonable to include a section on microbeam diffraction in any standard method.

Evaluation of Data Quality

Procedures for estimating the uniformity of the distribution of fibers on the filter, and for estimating fiber count confidence limits should be explained in detail in any method. There should also be a discussion of how QA procedures are related to reported values and to confidence limits. That is to say, procedures to take into account blank counts should be included in the analysis of errors. Such a discussion is lacking in most methods, and is given only cursory treatment in others, such as that by Yamate. Statisticians at NIOSH and/or NBS could potentially prove helpful in better defining procedures for evaluating data quality.

Ergonomics

The ergonomics of microscope operation (i.e., the fitting of the equipment and workstation layout to the person rather than vice-versa) are critical in determining both discomfort and fatigue during analysis. These factors are well known to affect fiber counts. It would therefore seem sensible to include some discussion of these factors in the TEM method, along with the usual discussion of the need for frequent breaks.

Operator Training and Certification

The obvious differences in operator skill and training made evident during the Peer Review session pointed up the need for more uniformity in training. Important aspects of this training should be covered in the Method. It is also clear that an operator certification program is badly needed to ensure that all microscopists using the TEM method are capable of certain minimum analytical skills. We therefore urge that a certification program be established as soon as possible.

4. Implementation

The foregoing suggestions, mostly for items that need to be spelled out or dealt with in a standard method, fall into two general categories: those that could be immediately included in a method, and those for which more research is needed before they could be written into a method. Table 1 shows which of these suggestions fits into one category or the other. Note that "Evaluation of Data Quality" is in the column on items that require further research. However, much of what would need to go into this section is already known. From the table it is evident that much can be done now. We therefore urge the adoption of a standard method containing these elements as soon as possible.

Table 1

Could be included in a method now	Need further research
1. Comparison to PCM disclaimed	1. Use of tortuous pore filters
2. Sampling details, assuming Nuclepore filters, and including grounding of conductive cassettes	2. Permissible magnification range
3. Contamination control	3. Evaluation of data quality
4. Length lower limit	
5. Low magnification scan	
6. Counting criteria with nomenclature	
7. Minimum instrument requirements	
8. Low dose techniques	
9. Microbeam diffraction	
10. Ergonomics	
11. Operator training	

APPENDIX A

PEER REVIEW OF ASBESTOS ANALYTICAL PROCEDURES

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1.0 BLANK DATA DISCUSSION - PROBLEM DEFINITION

Several recent EPA/WERL studies have been conducted to collect air samples before, during, and after asbestos abatement projects. Two analytical protocols were used to estimate airborne asbestos fiber concentrations: phase-contrast microscopy (PCM) and transmission electron microscopy (TEM). Samples for subsequent TEM analysis were collected on 37-mm polycarbonate (Nuclepore brand) filters in 3-stage plastic cassettes. The analytical protocol for the TEM samples that was specified in the study requirements was the method developed by George Yamate of IITRI.

The studies described herein were conducted by PEI Associates, Inc. for EPA/WERL during 1984 and 1985. Air samples were collected during various phases of asbestos abatement projects at three different buildings located in Corvallis, Oregon; Houston, Texas; and Columbus, Indiana. The population of air samples is differentiated into sample groups or individual "studies" according to sampling site location (i.e., Corvallis, Houston, or Columbus), date of sample collection, and the sample collection time with regard to the asbestos abatement project (i.e., postabatement, preabatement, etc.). Using these criteria, there are eight different sample groups (refer to Table 1).

Recent analytical data from several of the studies have shown: a) an undue amount of asbestos fibers present on study blanks; and 2) variation in the analytical results obtained by different laboratories. Table 1 summarizes the analytical results for 53 blank samples corresponding to the eight different sample groups. The results in Table 1 are reported as the number of asbestos, nonasbestos and ambiguous structures identified in 10 grid openings at 20,000 x magnification.

Apparent contamination of polycarbonate field blanks was first noticed during the analysis of the samples from Sample Groups 5 and 7 (Houston). This apparent contamination was characterized by the presence of an unusually large number of asbestos structures on the field blanks. These sample analyses

TABLE 1. SUMMARY OF FIELD BLANK TEM ANALYSES

					McCrone Structures/10 grid openings, 20,000X			NIOSH Structures/10 grid openings, 20,000X			Yamate Structures/20 grid openings 20,000X			ETC Structures/10 grid openings 20,000X		
Sample group	Date	Study	Sample No.	Location of carbon coating	As[a]	Nonas[b]	Amb[c]	As	NOID[d]	Amb	As	Nonas	Amb	As	Nonas	Amb
1	7/23 to 8/11/84	Columbus East Postabatement, Phase 1	EPA-15	EPA							2	0	0			
			EPA-104	EPA	2	0	0				2	0	0			
			EPA-105	EPA							0	0	0			
			EPA-106	EPA	1	0	0				6	0	0			
2	8/17, 8/18/84	Corvallis, Postabatement	COR-N-60	O.S.U.	1	0	0				0	0	0			
			COR-N-61	O.S.U.	1	0	0				1	0	0			
			COR-QA-N-1	NIOSH	30	0	0	<1[e]								
			COR-QA-N-2	NIOSH	44	0	0	<1[e]								
3	5/11 to 6/3/85	Columbus East Preabatement, Phase 2	EN-115	McCrone	16	0	0									
			EN-131	McCrone	15	0	0									
			E-CE-N-163	McCrone	16	0	0									
			E-CE-N-179	McCrone	38	0	0	6	6	7						
			E-CE-N-195	McCrone	10											
			E-CE-N-211	McCrone	16	0	0									
	/13 to 17/85	Columbus East Preabatement, Phase 2 (OTS)	EN-140	NIOSH				0	6	1						
4	6/30 to 6/9/85	Columbus East Postabatement, Phase 2	E-CE-N-722	McCrone	17	0	0	4[f]	4[f]	3[f]						
			E-CE-N-724	McCrone	21	0	0									
			E-CE-N-764	McCrone	12	0	0									
			E-CE-N-778	McCrone	29	0	0	13	0	25						
			E-CE-N-984	McCrone	21	0	0									
			E-CE-N-1003	McCrone	17	0	0									
			E-CE-N-1099	McCrone	14	0	0									
			E-CE-N-1109	McCrone	22	0	0									
			E-CE-N-1163	McCrone	12	0	0									
			E-CE-N-1173	McCrone	14	0	0									
			E-CE-N-1255	McCrone	31	0	0									
5	7/1 to 7/2/85	Houston Preabatement	E-HI-N-566	McCrone	10	0	0									
			E-HI-N-558	McCrone	3	0	0									
			E-HI-N-550	McCrone	10	0	0	3	5	7						
			E-HI-N-574	McCrone	9	0	0									
			E-HI-N-575	McCrone	28	0	0	3.1[e]								
			E-HI-N-576	McCrone	34	0	0	1.2[e]								

(continued)

TABLE 1 (continued)

Sample group	Date	Study	Sample No.	Location of carbon coating	McCrone Structures/10 grid openings, 20,000X			NIOSH Structures/10 grid openings, 20,000X			Yamate Structures/20 grid openings 20,000X			ETC Structures/10 grid openings 20,000X		
					As[a]	Nonas[b]	Amb[c]	As	NOID[d]	Amb	As	Nonas	Amb	As	Nonas	Amb
6	7/31 to 8/4/85	Columbus East - Mike Beard's QA Samples	E-CE-N-1174	NIOSH										9	9	
			E-CE-N-1175	NIOSH										0	0	
			E-CE-N-1176	NIOSH										3	1	
			E-CE-N-1177	NIOSH										5	9	
			E-CE-N-1204	NIOSH										1	1	
			E-CE-N-1205	NIOSH										25	6	
			E-CE-N-1206	NIOSH										0	8	
			E-CE-N-1207	NIOSH										1	0	
7	6/16 to 8/20/85	Houston Post-abatement	E-HI-N-1263	McCrone	3	0	0									
			E-HI-N-1271	McCrone	22	0	1									
			E-HI-N-1277	McCrone	42	0	0	5	11	6						
			E-HI-N-1287	McCrone	33	1	0									
8	11/18 to 11/22/85	Columbus East Postabatement, Phase 2 (OTS)	E-CE-N-1350	NIOSH				0	2	1						
			E-CE-N-1351	NIOSH				1	1	1						

---

[a] As = asbestos structures. Includes chrysotile and amphibole fibers, bundles, clumps, and matrices.

[b] Nonas = nonasbestos structures.

[c] Amb = ambiguous structures.

[d] NOID = no identification of structure.

[e] More than 100 grid openings were examined at 10,000X. Number shown indicates an estimate of the number of structures counted in 10 grid openings, assuming all structures to be asbestos. Breakdown on structure composition was not given.

[f] 95 grid openings were examined at 20,000X. Number shown indicates an estimate of the number of structures counted in 10 grid openings.

[g] Sample damaged by carbon coating.

were performed by the McCrone Laboratory. Contamination was suspected because the number of asbestos structures identified on the blank filters was higher (in most cases) than the number of asbestos structures identified on the exposed polycarbonate sample filters which had had air drawn through them for the specified sampling time. The results of all TEM analyses for the Houston sample groups (blanks and exposed samples) are summarized in Table 2. Blank correction of the exposed air samples (i.e., subtracting the average number of asbestos structures on the blanks from the number of asbestos structures on each exposed sample) was not feasible because a majority of the sample data points would be lost. Table 3 presents the result of blank correction on the Houston sample data.

This preliminary information regarding contamination of polycarbonate field blanks caused PEI and EPA/WERL to have all of the field blanks from Sample Groups 3, 4, 6, and 8 analyzed by transmission electron microscopy. These data are summarized in Table 1. Two of the blanks from Sample Groups 1 and 2 (which had previously been analyzed during 1984 and had shown no blank contamination problem) were also reanalyzed by the McCrone Laboratory. It was also considered prudent at this time to select blanks from various sample groups for subsequent analysis by TEM laboratories other than those of McCrone and Yamate. These additional analyses (shown in Table 1) were sought to confirm the large number of asbestos structures identified by the McCrone Laboratory. As shown in Table 1, there is considerable variation between the results of the sample analyses performed by different laboratories. In Figure 1, the geometric mean number of asbestos structures per sample is presented by analyzing laboratory and by sample group.

It is unclear at this time whether the apparent contamination is due to filter manufacture, filter cassette assembly, sample transport, sample preparation, sample analysis, or some other unknown factor. In an effort to gather more information regarding possible sources of blank contamination, six additional polycarbonate filters were submitted for analysis. Each polycarbonate filter was removed from its original container and cut into halves. Each filter half was then placed into a new (unused) Millipore plastic cassette assembly containing a back-up pad. Three filters (i.e., six plastic cassette assemblies each containing a polycarbonate filter half) were

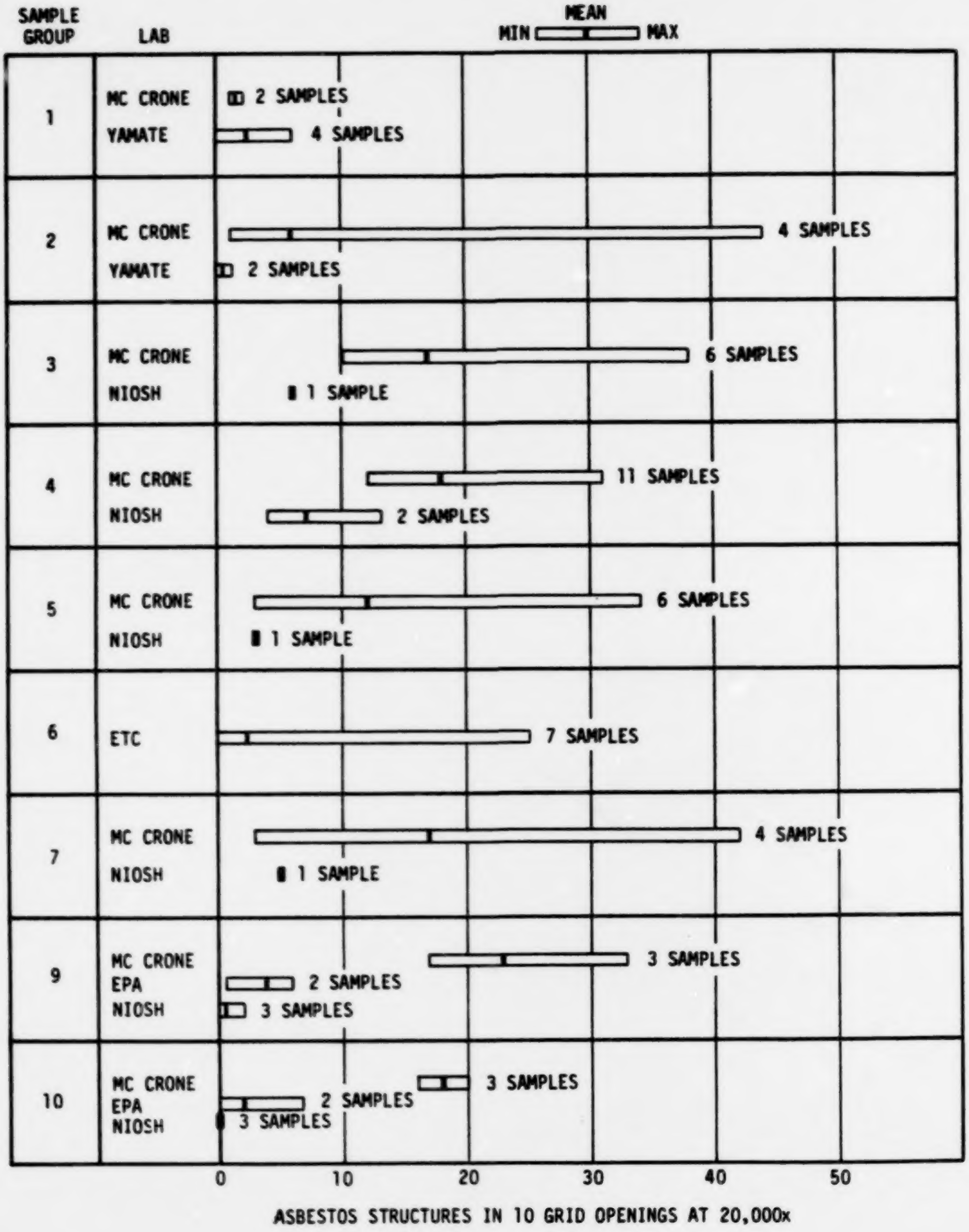

Figure 1. Comparison by laboratory of number of asbestos structures identified in blank samples.

TABLE 2. HOUSTON SAMPLE DATA

Preabatement						Postabatment					
Nonaggressive			Aggressive			Nonaggressive			Aggressive		
Sample no.	No. of asbestos structures/10 grid openings, 20,000x	Concentration, 10^6 asbestos structures/m^3	Sample no.	No. of asbestos structures/10 grid openings, 20,000 x	Concentration, 10^6 asbestos structures/m^3	Sample no.	No. of asbestos structures/10 grid openings, 20,000 x	Concentration, 10^6 asbestos structures/m^3	Sample no.	No. of asbestos structures/10 grid openings, 20,000x	Concentration, 10^6 asbestos structures/m^3
543	7	0.050	551	33	0.210	1274	18	0.140	1279	35	0.250
544	23	0.160	552	11	0.070	1275	17	0.140	1280	22	0.160
545	7	0.050	553	10	0.065	1278	10	0.078	1281	21	0.150
546	21	0.160	555	17	0.120	1257	24	0.190	1265	6	0.044
547	19	0.140	556	26	0.180	1258	15	0.120	1266	8	0.059
548	14	0.100	557	7	0.048	1259	35	0.270	1267	8	0.058
559	19	0.130	568	37	0.260	1283	17	0.130	1288	20	0.150
560	35	0.240	569	13	0.091	1284	20	0.160	1289	39	0.280
561	12	0.084	570	28	0.200	1285	30	0.250	1290	23	0.180
563	5	0.033	571	17	0.130	1260	25	0.210	1268	14	0.100
564	11	0.073	572	35	0.260	1261	8	0.065	1269	19	0.140
565	23	0.150	573	16	0.120	1262	15	0.120	1270	32	0.230
Ambient						Ambient					
549	23	0.180				1264	0	<0.009			
554	5	0.036				1272	21	0.190			
562	13	0.091				1276	27	0.220			
567	39	0.310				1282	20	0.190			
						1286	26	0.230			
						1291	28	0.300			

TABLE 3. HOUSTON DATA - BEFORE/AFTER BLANK CORRECTION[A]

Sample type	Number of Sample Results Before Blank correction	Number of *Viable Sample Results after blank correction	Number of data points lost
Preabatement-nonaggressive	12	9	3
Preabatement-aggressive	12	11	1
Ambient-outdoor	4	3	1
Postabatement-nonaggressive	12	2	10
Postabatement-aggressive	12	3	9
Ambient-outdoor	6	3	3

*Viable = Zero or positive numbers

[A] Preabatement samples corrected for the average of the preabatement blanks - or (8 asbestos structures subtracted from original data)

Postabatement samples corrected for the average of the postabatement blanks (or 26 asbestos structures subtracted from original data)

31

prepared at the EPA facilities in Cincinnati. Three more filters were prepared at the Cincinnati PEI facilities. Six split filter samples were either mailed or hand-carried to two TEM laboratories; the McCrone Laboratory and the NIOSH Laboratory in Cincinnati. (Note: Samples at the NIOSH facility were analyzed by a PEI TEM analyst using NIOSH microscopy equipment and with the assistance of NIOSH personnel). Table 4 presents the TEM analytical results for these split filter samples. At the collaborative technical meeting held April 24 and 25, 1986, the peer reviewers recommended that additional TEM analyses of these split filter samples be conducted. Additional analyses were performed on some of the samples by Energy Technology Consultants (ETC). These data are also summarized in Table 4.

2.0 SAMPLE CASSETTE PREPARATION

Preparation of the sample cassettes used in these air sampling studies was performed in the following manner. Nuclepore polycarbonate membrane filters (0.4-µm pore size, 37-mm diameter) were obtained from EPA-WERL Cincinnati, and the manufacturer's lot number of the packaged filters was recorded. With each lot, the polycarbonate filters were packed by the manufacturer in plastic boxes containing 100 filters per box. These boxes were not numbered by the manufacturer, nor labelled in any special manner during these air sampling studies. The polycarbonate filters were stored at the EPA facilities and obtained by PEI on an as-needed basis. New, preassembled, 37-mm diameter, three-stage plastic cassettes were purchased by PEI from Millipore Corporation or obtained from EPA-WERL. These cassettes were stored in their original packages at PEI or EPA and used as needed. Cellulose ester membrane filters (5.0 µm pore size, 37-mm diameter) for use as backup pads were purchased from the Millipore Corporation.

Polycarbonate filters were placed in the plastic sample cassettes at the PEI facilities located in Cincinnati, Ohio. Sample cassette preparation was performed by several PEI technicians or industrial hygienists in clean office areas. The building in which the PEI offices are located has been inspected for the presence of asbestos-containing building materials. No asbestos-containing building materials were found in the facility. Asbestos bulk and

TABLE 4. SUMMARY OF TEM ANALYSES BY LABORATORY FOR SIX NON-FIELD BLANKS

Date	Study	Sample no.	Location of carbon coating	McCrone Structures/ 10 grid openings 20,000x			PEI/NIOSH[a] Structures/ 10 grid openings 20,000x			EPA-Duplicates[b] Structures/ 10 grid openings 20,000x			EPA-Prepared[c] Structures/ 10 grid openings 20,000x			ETC Structures/ 10 grid openings 20,000x		
				As[d]	Nonas[e]	Amb[f]	As	NOID[g]	Amb	As	NOID	Amb	As	NOID	Amb	As	Nonas	Amb
2/86	Samples assembled by W. Cain, EPA	W-1-A	NIOSH				0	21	1	5	18	2	1	12	6			
		W-1-B	McCrone	22	0	0										6	1	0
		W-2-A	NIOSH				9	3	3	3	22	4	7	11	2			
		W-2-B	McCrone	33	0	0										3	1	0
		W-3-A	NIOSH				2	8	1									
		W-3-B	McCrone	17	0	2										15	5	0
2/86	Samples assembled by A. Crone, PEI	P-1-A	NIOSH				0	16	0	5	16	2	0	10	0			
		P-1-B	McCrone	20	0	0										15	0	0
		P-2-A	NIOSH				0	16	2	1	32	0	7	14	1			
		P-2-B	McCrone	16	0	0										0	0	0
		P-3-A	NIOSH				0	14	2									
		P-3-B	McCrone	19	0	0										6	0	0

[a] Samples analyzed by a PEI microscopist using NIOSH microscopy equipment with the assistance of NIOSH personnel.

[b] Duplicate analyses of the PEI/NIOSH sample grids (shown in the preceding column). Samples grids prepared by PEI/NIOSH, analysis performed by EPA microscopist.

[c] Replicate analysis of the previously carbon-coated filter sample. Sample grid preparation and analysis performed by an EPA microscopist.

[d] As = asbestos structures. Includes chrysotile and amphibole fibers, bundles, clumps, and matrices.

[e] Nonas = nonasbestos structures.

[f] Amb - ambiguous structures.

[g] NOID = no idenfication of structure possible.

air samples are analyzed by the PEI Environmental Laboratory located in the basement of the building. However, no sample cassette assembly was performed near the laboratory facilities, nor by laboratory personnel.

The sample cassette assembly sequence that was followed included the following steps:

1. A desk or table located away from drafts and ventilation grills was wet-wiped with dust-free cloths dampened with isopropyl alcohol or a soap and water solution.

2. After removing the facecap (top section) of a clean, three-stage cassette, a pair of cleaned forceps was used to remove the 0.8-μm cellulose ester membrane filter. This filter was discarded and replaced with a new 5.0-μm cellulose ester filter.

3. A polycarbonate filter was removed from the manufacturer's box and placed in the plastic cassette on top of the 5.0-μm cellulose ester filter and backup pad.

4. The facecap of the plastic cassette was replaced.

5. Electrical tape (nonasbestos, PVC composition) or a cellulose shrink band (Gelbrand) was placed around the outside of the closed, three-stage plastic cassette to seal possible points of leakage.

6. The outside of each sample cassette was numbered using a felt-tipped marker.

7. Numbered and sealed sample cassettes were returned to the original Millipore box. Sample cassettes were stored in these boxes until actual air sampling was performed.

Extreme caution was used during preparation of the sample cassettes to ensure that filters, cassettes facecaps, and forceps did not contact work surfaces or clothing.

APPENDIX B

NUCLEPORE® MEMBRANE FILTERS

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Pleasanton, California 94566-3294

I. Membrane Filtration - separation of particles in a liquid or gas by a defined pore structure fabricated into a thin, synthetic membrane filter (< 200um). - Figure 1.

II. Types of Membrane Filtration

1. Reverse Osmosis (RO) - separation of dissolved ionic species - typically with retentive pores of less than 10 Å (100 nonometers, 0.001um). Associated, characteristically, with high pressures (> 200 psi) because you have to exceed the osmotic pressure.

2. Ultrafiltration (UF) - separation of dissolved macromolecules (e.g. proteins) and colloidal species with retentive pores of 10 to 1,000 Å (0.001 to 0.1um). Typical pressures used to effect separation are 10 to 125 psi.

3. Microfiltration (MF) - separation of particles with retentive pores of 1,000 Å to 100,000 Å (0.1 to 10um). - Figure 2.

III. Types of Membrane Filters

1. Depth or tortuous pore - rated by the largest effective pore (e.g. 0.22um retains all larger than). - Figure 3.

2. Screen or capillary pore - rated by actual pore definition. (e.g. 0.2um is actual diameter of pore - + 0, - 20%). Nuclepore polycarbonate and polyester membranes are the present, commercial examples. - Figure 4.

   a. Polycarbonate - 0.01 to 14.0um.
   b. Polyester - 0.1 to 12.0um.

IV. Manufacture

1. Depth (tortuous pore) - solvent casting technique. Casting solution composition, temperature of solution and atmospheric

conditions and casting technique determines pore size, pore density (porosity), thickness, etc. - Figure 5. See structure in Figure 6.

2. Screen (capillary pore) - track etch technique.

   a. So called because a thin film (polcarbonate or polyester) is bombarded with fission fragments, under controlled conditions, leaving damage tracks on the film. After aging (cooling down), the irradiated film is etched in a controlled temperature caustic bath which develops the pores. In actuality, the thickness of the film is etched away to the same degree as to the developed pore diameter (e.g. 0.4um pore, 0.4um less film thickness). - Figure 7 and 8.

   b. Steps in process.

      (1) Film - purchased from supplier.
      (2) Film rewound at Nuclepore into usable configuration.
      (3) Film shipped to irradiator (Univ. of Missouri Reactor facility - Nuclepore owns insert unit and manages the actual operation pertaining to Nuclepore membranes).
      (4) Irradiation of film.
      (5) Shipment of irradiated film to Nuclepore - Pleasanton.
      NOTE: Special, heavy containers are used for shipping.
      (6) Aging of film in Pleasanton, CA (cooling down).
      (7) Etching of irradiated film in caustic solution.
      (8) Neutralization of caustic on film.
      (9) Rinsing of etched film with deionized water containing alcohol (bacteriostat).
      (10) Drying of film (Hepa filters used).
      (11) Packaging
         (a) Cut and sandwiched into wraps (separator parchment paper between sheets).
         (b) Cut into final disc or sheet size.
         (c) Boxed and shrink - wrapped.
      (12) Submitted to finished goods inventory pending Q.A. approval.
      (13) Shipped to customer.

V. Example of Asbestos on Nuclepore Polycarbonate Membrane Filter. - Figure 9.

Ben Carter

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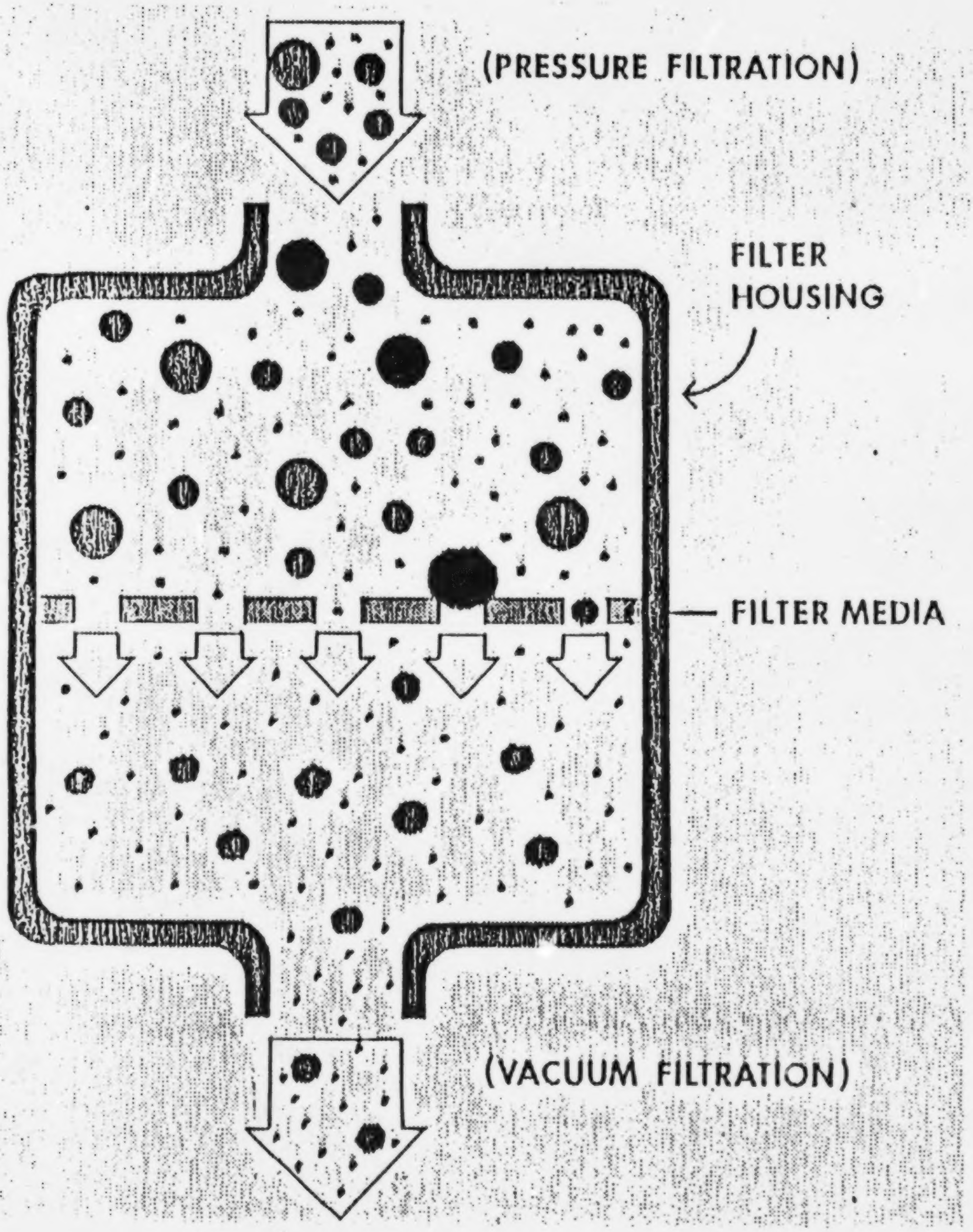

(PRESSURE FILTRATION)
FILTER HOUSING
FILTER MEDIA
(VACUUM FILTRATION)

FIGURE 1

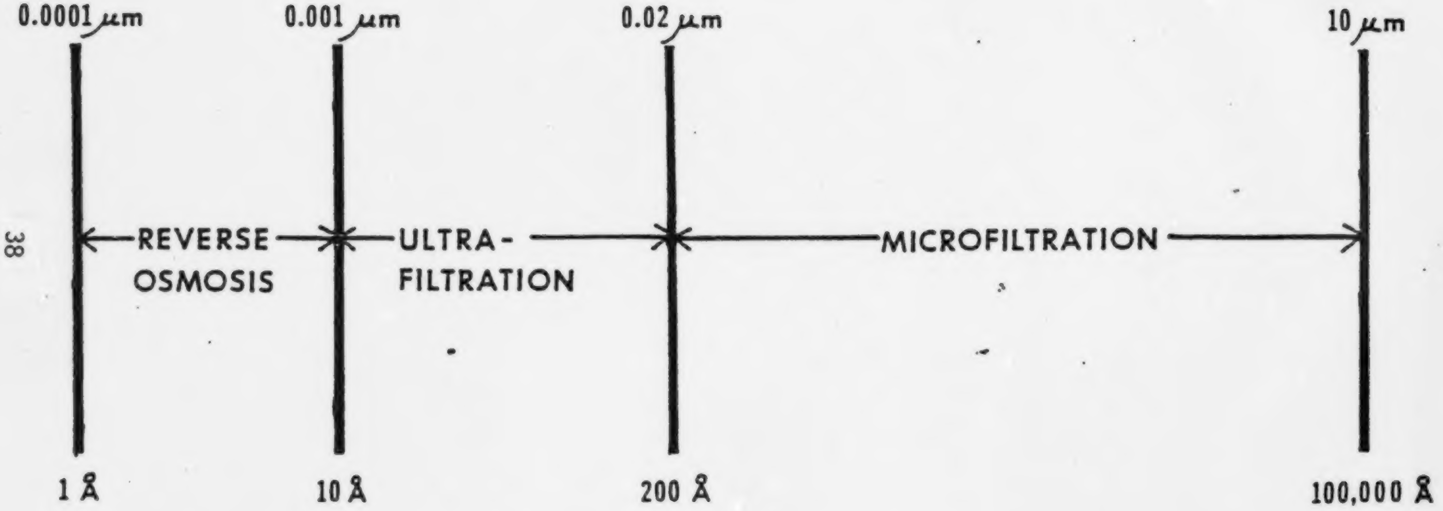
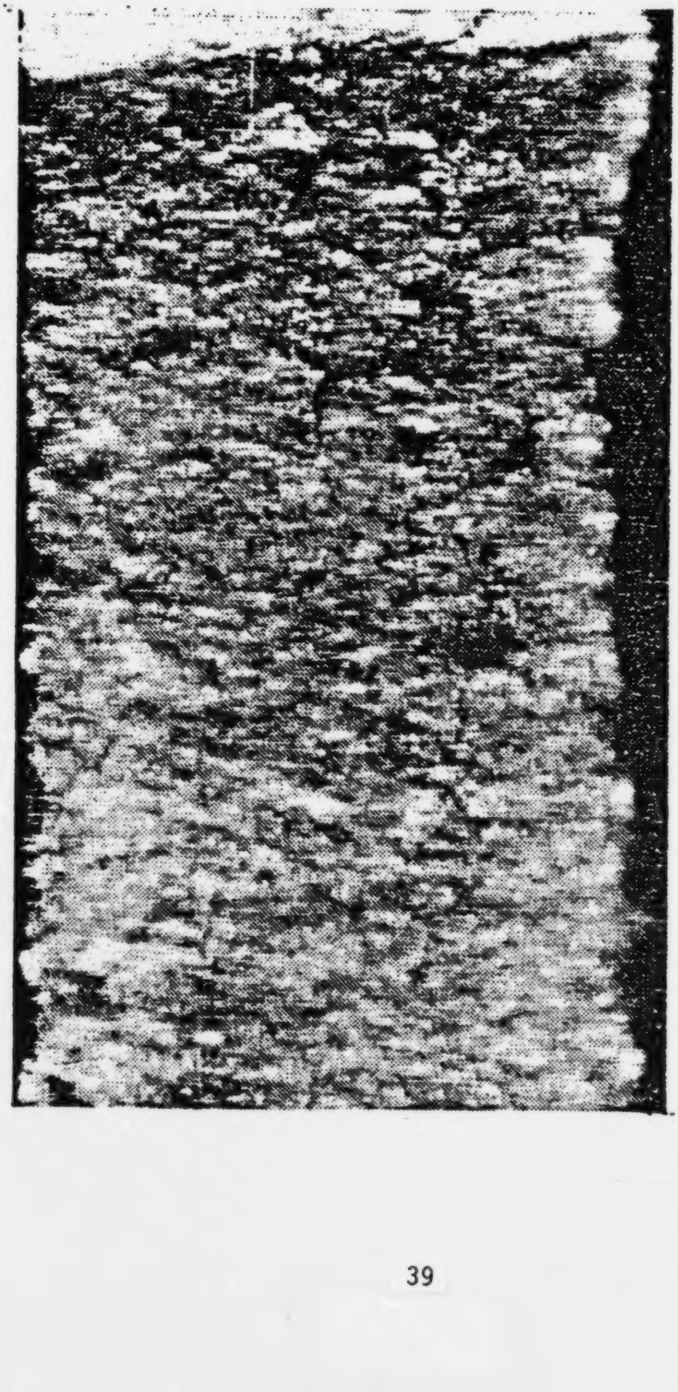

0.0001 µm
0.001 µm
0.02 µm
10 µm
REVERSE OSMOSIS
ULTRA-FILTRATION
MICROFILTRATION
1 Å
10 Å
200 Å
100,000 Å

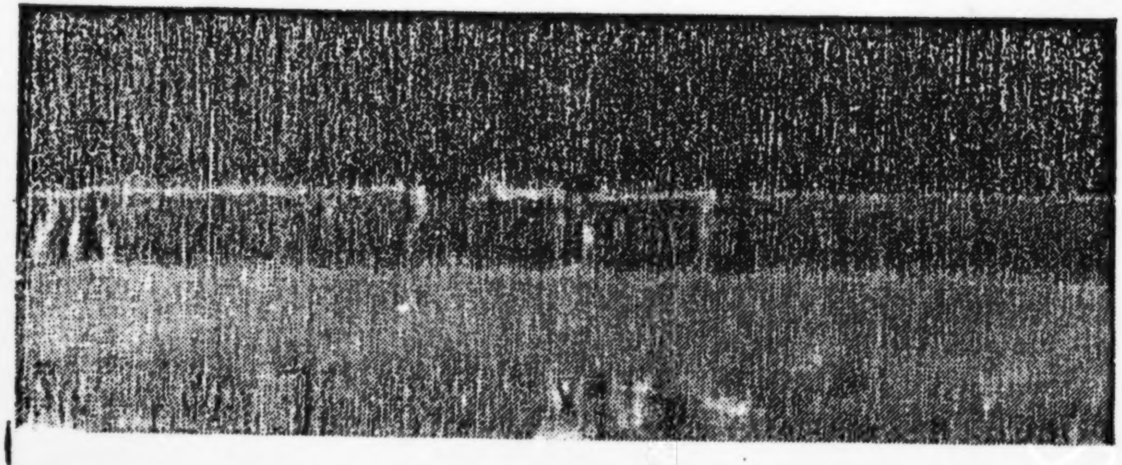
FIGURE 2

Reproduced from best available copy.



Cross section of depth membrane (5μm pore size) magnified 8,000X.

FIGURE 3



Cross section of screen membrane (8μm pore size) magnified 15,000X.

FIGURE 4

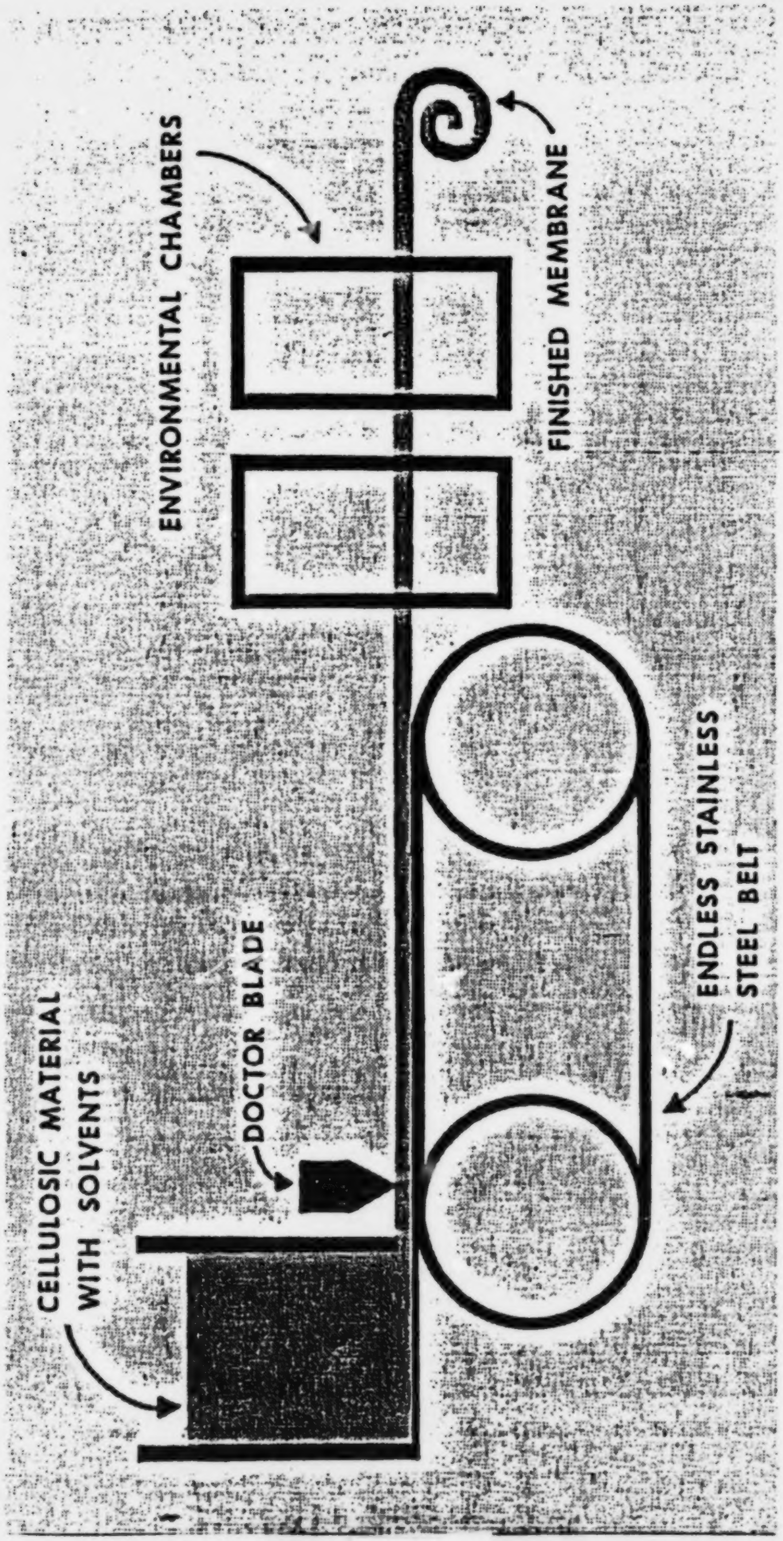
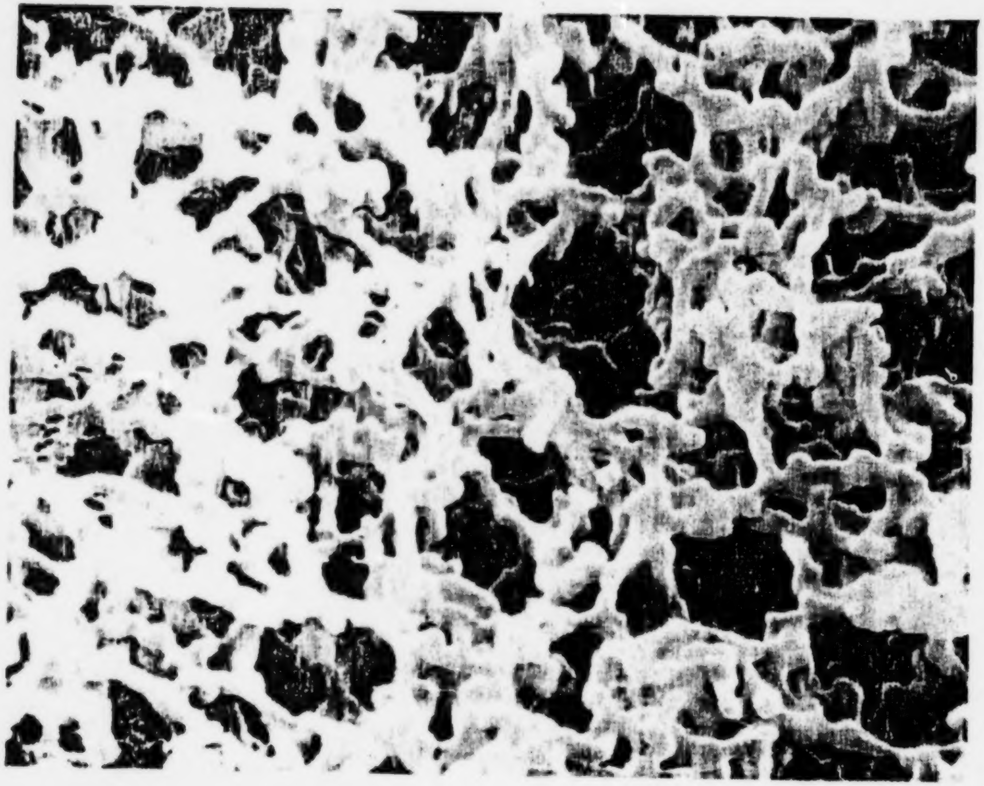
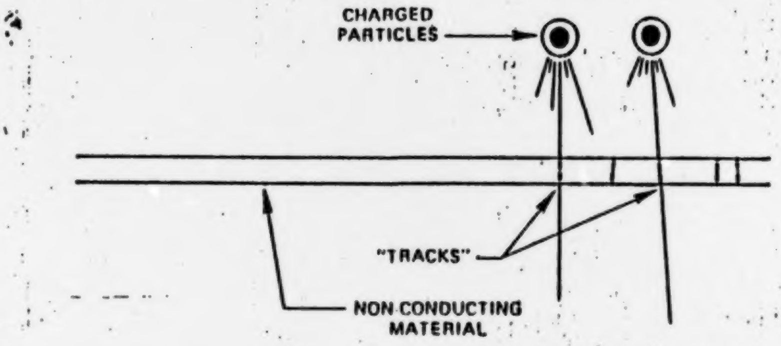

CELLULOSIC MATERIAL WITH SOLVENTS
DOCTOR BLADE
ENDLESS STAINLESS STEEL BELT
ENVIRONMENTAL CHAMBERS
FINISHED MEMBRANE

FIGURE 5

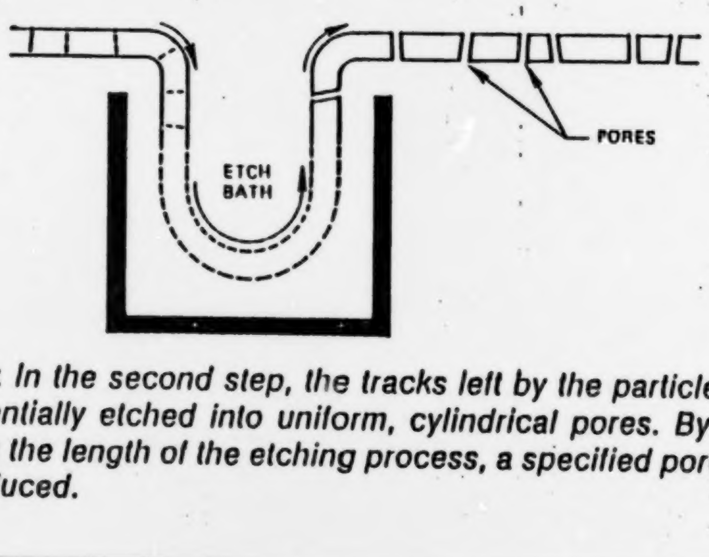


(Magnified 10,000X)

FIGURE 6


CHARGED PARTICLES
"TRACKS"
NON CONDUCTING MATERIAL

Step 1: *In the Nuclepore two-step process, polycarbonate film is first exposed to collimated, charged particles in a nuclear reactor. As particles pass through the material, they leave sensitized tracks. Pore density (pores/cm²) is controlled by the residence time in the irradiator.*


ETCH BATH
PORES

Step 2: *In the second step, the tracks left by the particles are preferentially etched into uniform, cylindrical pores. By controlling the length of the etching process, a specified pore size is produced.*

FIGURE 7

(Magnified 10,000X)

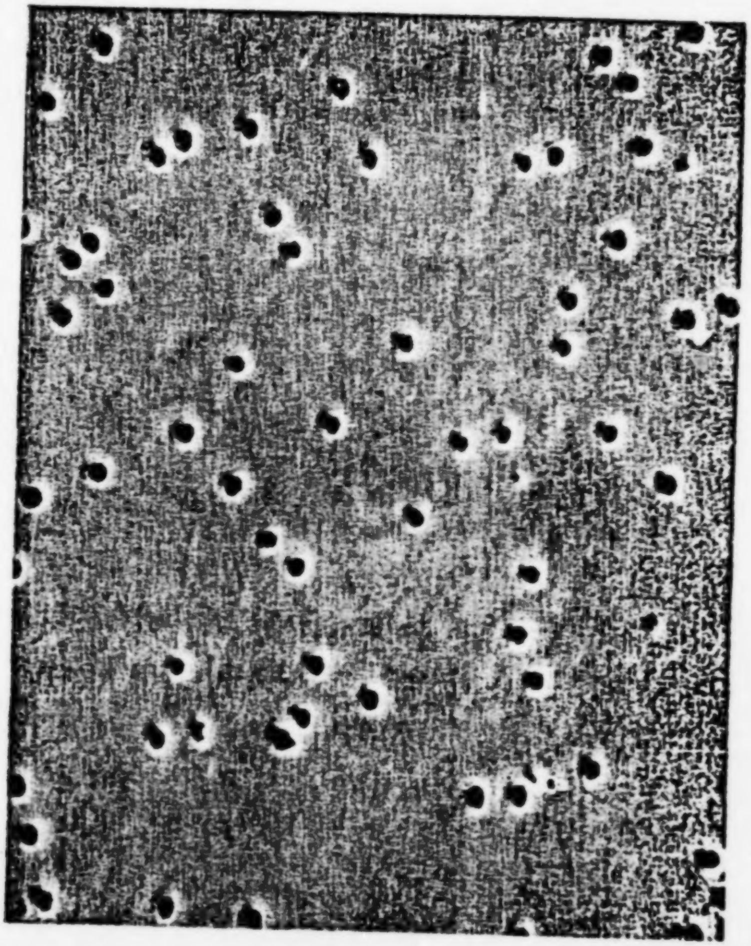


FIGURE 8

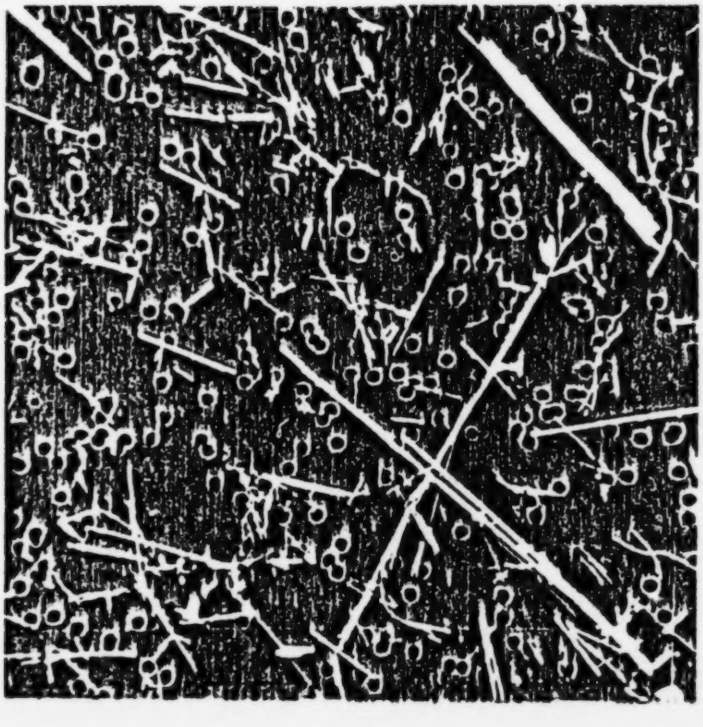


FIGURE 9

APPENDIX C

MILLIPORE'S MANUFACTURING PROCEDURES FOR AEROSOL MONITORING PRODUCTS

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Linda Dohrman
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NOTE: The editor requested that the Millipore Corporation provide additional information on the quality assurance employed during manufacture of their cellulose ester filters, which in some asbestos samplings have been substituted for standard polycarbonate filters.

General

The Millipore Corporation is a high technology, multinational corporation that markets products for the analysis and purification of fluids. It serves a technical customer base, including research scientists, production engineers, quality control professionals and physicians. Its products are used in critical applications, where high analytical sensitivity or high purity is needed.

Manufacturing Process

Millipore's manufacturing facilities meet or exceed Medical Device GMP standards as a result of voluntary compliance. The filter material used in the aerosol monitors is produced from raw materials that are rigidly inspected and controlled throughout the manufacturing process.

Each shipment of incoming materials or internally manufactured membrane is given a control number and quarantined until the required series of tests and evaluations is completed. The test results are recorded and become part of the lot history records. Incoming evaluations may include any or all of the following: visual inspections, chemical analysis, physical evaluations, biological testing, process compatibility evaluations, and certification review.

Approved raw materials are then processed into filter material using equipment specifically designed to minimize particulate and fiber contamination. Each roll of filter stock used in the aerosol monitor is sampled for the particulates and fibres by an independent laboratory using the NIOSH Method #7400 counting Rule B.

During manufacturing all process components are strictly controlled and documented assuring that reliability and performance are built into the product.

As an example, all water coming in contact with the product during manufacturing is pre-treated with ultra filtration and routinely tested for both bioburden and pyrogens. This results in a product with low trace metals.

In addition, no asbestos containing materials or equipment are used during the manufacture of these products.

In-process controls and final inspection of finished product confirm that the product has achieved all performance and quality specifications. As an example, each lot of aerosol monitors is subjected to fibre and particle count testing to confirm quality assurance and quality control.

Production history records are kept for every lot. These detail the raw material criteria, lot numbers, manufacturing, and test results.

APPENDIX D

LAB PROCEDURES

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I. INTRODUCTION

A. Necessity for Q.C. in Asbestos Analysis

1. Confidence and Reliability of Results
2. Strong as its Weakest Link

B. Q.C. in the Field

1. Record-Keeping
    - a. Logbook
    - b. Labelling
2. Sampling Procedures
    - a. Calibration of Pumps and Flow Rate
    - b. Filters and Cassette Loading
    - c. Field Blanks and Duplicates

C. Q.C. in the Laboratory

1. Documentation of the Lowest Level of Fibers Routinely Reported
    - a. Use of Field and Lab Blanks
    - b. Coded Samples
    - c. Duplicate Analysis
    - d. Shared (split) Analysis with Second Lab to Spot Check Accuracy of Results
2. Instrumentation
3. Analytical Procedures

D. Personnel Classification

1. Preparation
2. EM Analyst
3. Data Reduction and Report Responsibility

II. INSTRUMENTATION

A. Location and Physical Condition

1. Prep Room
2. EM Room

B. Instrument Log

1. Problems and Maintenance
2. Magnification Calibration

III. ANALYTICAL PROCEDURES

A. Sample Receipt

1. Chain of Custody or Contractual Samples by Quality Assurance Officer (QAO)
2. Routine Samples by EM Staff
3. Information Logged
    - a. Date of Receipt
    - b. Number of Samples
    - c. Sample I.D. (labelling checked)
    - d. Condition of Sample (from visual exam)
    - e. Add on IITRI I.D.
4. Safe Storage
    - a. Clean Bench
    - b. Locked During Non-Working Hours

B. Sample Preparation

1. Project Log -- Recheck Labels (I.D.) and Filter Surface
2. Vacuum Evaporator -- Started at Least 1/2 hour Before Use
3. Jaffe Washer Prepared while Evaporator Pumps Down
4. Samples to be Carbon-Coated
    - a. For 25 mm Cassettes - 7 can be prepared at once; 6 real and 1 lab blank
    - b. For Slides - Maximum of 2 Slides - 4 filter sections per Slide
    - c. For 37 mm - 3 samples
    - d. Possible Point of Contamination - during Placement and Removal from the Bell Jar
5. Grid Transfer Procedure
    - a. Grids on Ross Lens Tissue
    - b. Petri Disk (ground glass)
    - c. Foam
    - d. Filters
    - e. Screen
    - f. Solvent - Spectro-Quality for G.C.
    - g. Labelling of Grids on Petri Disk and Logbook
    - h. Grids Transferred to Grid Box and Locations noted in Logbook

6. EM Examination - Point of Contamination when Placed in Sample Holder and into EM

IV. PERSONNEL QUALITY CONTROL

A. Objective Evaluation

1. NBS Standard
2. Finder Grid
3. Split Samples

B. Subjective Evaluation

1. Attitude is Most Important
2. Treatment and Consideration

V. GUIDELINES

A. Central Filing of Blank Results

B. Order of Analysis

1. Field Blank, if clean go to regular samples
2. If 5 or more asbestos structure, per 10 grid openings, check lab blank

C. Lab Blanks to be Prepared with Each Set of Samples and Analyzed if no field blanks were designated

D. Test at Least one filter from each box even if several boxes have the identical lot number. If time is available, analyze a filter from the top and one near the center

E. Do not report results of 5 or more asbestos structures per 10 grid openings until the lab blank for the set had been analyzed.

VI. BASIC ASSUMPTIONS

A. Instrument is in Top Running Condition

B. Laboratory is Dedicated to Asbestos Analysis

C. Personnel is familiar with the use of the electron microscope and its imaging capabilities

D. Availability of Knowledgeable Support Personnel (In-House or Outside Source)

A SIMPLIFIED TEM ANALYSIS METHOD FOR ASBESTOS ABATEMENT PROJECTS[1]

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Abstract

Recently the Office of Toxic Substances, U.S. Environmental Protection Agency, revised the document "Guidance for Controlling Friable Asbestos-Containing Materials in Buildings" with the 1985 edition entitled, "Guidance for Controlling Asbestos-Containing Maaterial in Buildings" (EPA 560/5-85-024, June 1985).

Air monitoring has a well-defined role in determining when an abatement project is completed. The transmission electron microscopy (TEM) method, although technically superior, has cost, turnaround time, and instrument availability as disadvantages when compared to the Phase Contrast Microscopy (PCM) method with its inherent limiations.

This paper condenses the TEM method that was developed to cover a broad spectrum of asbestos analysis problem situations. That method has been streamlined within the abatement requirements of the 1985 edition. Data necessary for clearance standards are collected, while extraneous information is not.

The applications of the simplified method plus changes such as filter size, carbon coating, counting criteria, and scheduling are presented. Data and results obtained are also shown. Although the method still will not compare in time and cost with the PCM method, the gap has been reduced and thus provides a better option for prospective abatement work.

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[1] This paper was presented at the 1986 EPA/APCA Symposium on "Measurement of Toxic Air Pollutants" April 27-30, 1986. Raleigh, NC

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A SIMPLIFIED TEM ANALYSIS METHOD
FOR ASBESTOS ABATEMENT PROJECTS

Introduction

Asbestos is a generic term applied to several commercially useful fibrous silicate minerals and categorized as chrysotile asbestos and amphibole asbestos. The unique properties of asbestos, such as heat resistance, strength, chemical resistance, and morphology led to its extensive use in the construction industry. Sprayed-on or troweled-on asbestos-containing materials were commonly used in fireproofing and insulation of the structural components of a building. Asbestos was also incorporated in pipe and boiler room insulation, floor tiles, and decorative wall coverings.

The potential for injury by respiratory exposure to asbestos fibers has been well documented. Asbestosis, lung cancer, and mesothelioma (an incurable cancer of the pleura) have been attributed to asbestos inhalation and the numbers of these cases have been increasing yearly. The latency period for these asbestos-related diseases has been projected at 20 to 40 years, which makes it difficult to assess dose-response relationships, threshold values, or size/shape effects.

Since the U.S. Environmental Protection Agency (EPA) banned the use of asbestos-containing material (ACM) in these construction activities, the problem remained as to what to do with the ACM already in place. The EPA established a Technical Assistance Program (TAP) to provide technical expertise on how to identify and control ACM. The presence of asbestos in schools, public-access buildings, and commercial buildings required identification (location) and its condition (risk). Guidance documents were prepared and distributed in 1979--Orange Book,[1] in 1983--Blue Book,[2] and 1985--Purple Book,[3] which incorporated state-of-the-art results from numerous EPA sponsored studies.

A high risk situation contains deteriorating asbestos designated friable (when dry, could be easily crumbled, pulverized, or reduced to powder by hand pressure) in a location where many individuals would congregate for extended periods of time. Since children have the greatest potential for long term exposure, schools were the first choice objective in asbestos abatement. Building tenants and occupants were also in the risk group.

In asbestos abatement, the owner or manager has already surveyed his building and is aware of the presence, condition, and relative quantity of ACM. The building owners or managers, in considering the health and welfare of their occupants, are also aware of possible legal consequences and public reaction. At present there are four options for the responsible owner/manager. These are:

1. remove the ACM
2. encapsulate the ACM
3. enclose the ACM
4. establish a sound operation and maintenance plan.

The guidance documents provide to the owner/manager a course of action for controlling the ACM.

Asbestos abatement is a growing industry, with a concurrent increase in rules and regulations at the Federal and State levels. The Occupational Safety and Health Administration (OSHA) maintains responsibility for activities inside the enclosed abatement workplace. The EPA responsibility resides outside of the enclosure, checking for asbestos leakage and most important, whether the clean-up of asbestos in the enclosed air space is adequate for removal of the enclosure barriers without release of additional asbestos to the environment. State agencies responsible for asbestos abatement usually incorporate Federal rules, regulations, and guidelines into their regulatory procedures, thus enlarging the area of enforcement.

The EPA presented its latest findings in the 1985 document entitled, "Guidance for Controlling Asbestos-Containing Materials in Buildings," the so-called Purple Book.[3] In this document, release of the abatement contractor is based on visual inspection and air testing to determine that the source of asbestos fiber release has been controlled and the asbestos generated during the abatement activity is below a designated level of acceptance.

Three microscopic methods are used in airborne asbestos analysis. These are phase contrast microscopy (PCM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

The TEM method is the state of the art in identifying and quantifying the asbestos concentration levels in ambient air. It gives the most complete information on airborne asbestos since it can detect the extremely small and thin fibers as well as distinguish asbestos from other fibers. However, it has the reputation of being expensive ($200 to $600 per sample) and time-consuming (two- to seven-day turnaround), and the instrumentation is not widely available.

This presentation is made to show that in air testing for asbestos abatement projects, the recommended, comprehensive TEM analysis method developed under contract to EPA can be simplified. Using the guidelines of the Purple Book and without any sacrifice of quality, the method will result in a less costly and time-consuming analysis. The increased use of TEM analysis should result in a corresponding increase in the number of TEMs.

Method Development

Background

The EPA sponsored an in-depth study of various electron microscopy procedures that resulted in the provisional methodology manual, "Electron Microscope Measurement of Airborne Asbestos Concentrations."[4] Limitations in the provisional method resulted in a follow-up study and the development of a refined transmission electron microscope method for the determination of asbestos in the ambient environment. The refined method, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy,"[5] incorporates the basic concept of the provisional methodology as one of its protocols. However, both methods are referenced in the Purple Book for air testing for abatement clearance.

The refined methodology is divided into three levels of data acquisition. Level I is a relatively rapid procedure and provides information on fiber number, size distribution, visual selected area electron diffraction (SAED) pattern, and a calculated mass concentration. Level II adds elementary chemical analysis for individual fibrous structures, and Level III confirms asbestos identification by quantitative electron diffraction pattern analysis.

To meet the objectives of the method's development, emphasis was placed on eliminating non-essential parts of the Level I analysis, incorporating time-saving features of data acquisition and reduction, and changing to a smaller filter size in the air sampling equipment.

Non-Essential Features

The first feature eliminated was the size measurement of the individual fibrous structures. Measurement required positioning of the structure relative to concentric circles of known radius, taking length and width estimates, and finally repositioning back to its original location in order to continue traversing across the grid opening. The writing of lengths and widths onto the data sheet also added to the time factor. Once voice-activated input to a computer is perfected, this feature may be added back to the method.

The second feature to be eliminated was the transfer of data into the computer for a computerized printout of the results. The computer generates calculated mass of individual fibers, aspect ratios, average dimensions, etc. However, according to the Purple Book, only the number of fibers per cc is of importance, and it is used to compare inside and outside levels of the enclosed abatement area. Therefore, the data sheet can be simplified without concern for computer input requirements.

The third feature to be eliminated was the cumulative fibrous structure classification, since in asbestos abatement clearance testing the concentration levels inside the enclosure are monitored during the workday with phase contrast microscopy. Asbestos levels are minimal when clean-up is completed.

Time-Saving Features

The data sheet for the electron microscopist has been redesigned. All pertinent information that was included in the Level I data sheet, computer printout, and report form were incorporated into the abatement data sheet that would be filled in and calculated by hand. The results themselves would then be reported directly to the requestor of the analysis (asbestos project manager); the data sheet would be kept on file.

Change to 25 mm Filter Size

The TEM methods have in the past recommended the 47 mm and 37 mm filter sizes. A change to the 25 mm size results in several time and cost savings.

1. A smaller sampling volume would be required for the same detection limit, or a lower detection limit would be attained for the same sampling volume, as shown in Table I.
2. With the use of a smaller size filter, more filters can be carbon-coated at one time.

3. The cost of 25 mm filters is less than half that of the 37 mm size. The possibility of a box of filters being contaminated exists, and replacement costs would not be as great. The other filter cassette components would be correspondingly lower in cost. Although field samples and field blanks would be supplied by the asbestos project manager, each laboratory has to prepare its own laboratory blanks.

Results (Simplified Method)

This simplified TEM asbestos analysis method is specifically designed for air testing in an asbestos abatement project and includes the following steps:

1. The 25 mm, 0.4 μm polycarbonate filters in a box are spot checked for asbestos contamination prior to use of the remainder.
2. The three-piece filter cassettes are loaded on a clean bench. A support pad is placed into the cassette base, then a 5.0 μm pore size cellulose ester backing filter is placed on the support pad. Finally, a clean 0.4 μm polycarbonate filter with the shiny side up is placed on the backing filter. The cassette assembly is pressed tightly together to hold the filters in place and prevent leaks from the sides of the cassette. An added precaution is to use a plastic tape to seal the sides. The assembly is then placed in a plastic bag to keep it dust-free until needed.
3. A known volume of air is passed through the polycarbonate filter to obtain approximately 5 to 10 μg of particulates per cm^2 of filter surface. Knowledge of the particulate loading of the source of make-up air into the abatement enclosure will aid in the selection of the sampling volume.
4. Periodic flow rate checks are made with a calibrated flow meter.
5. Upon completion of sampling, the cassette is removed while upright, placed carefully into a plastic bag, and placed in the carry case and hand-carried back to the laboratory.
6. The filter is carbon-coated while still in the cassette by removing only the top lid.
7. The particulates are transferred to an electron microscope (EM) grid using a refined Jaffe Wick Washer.
8. The EM grid is examined under low magnification (250X and 1000X), followed by high magnification search and analysis (20,000X or approximately 16,000X on the fluorescent screen).
9. A known area (measured grid opening) is scanned and the fibrous structures (fibers, bundles, clusters, and matrices) are identified as to asbestos, ambiguous, or non-asbestos; length measured for categorization; and counted. No distinction is made between chrysotile and amphibole asbestos.
10. The observations are recorded for 10 grid openings on the data sheet. For better statistics, another 10 grid openings in a second grid from the same filter may be examined. Figure 1 is an example of a completed data sheet.

Since rates per hour vary between personnel, a standard of comparison may be the analytical time necessary to complete a filter analysis. Previously, a Level I analysis took 3 to 4 hr per sample. With the new method it takes 2 to 2-1/2 hr to complete an analysis.

Thus, for an abatement clearance test, if samples arrive by 3:00 P.M., carbon coating and grid transfer is started before 5:00 P.M. The next morning by 9:00 A.M., samples could be examined in the electron microscope. Results could be telephoned out to the job site 40 hr after receipt of the samples.

Conclusions

A simplified TEM analysis method for asbestos abatement projects is described. The important data necessary for proper evaluation of the airborne levels of asbestos in the abatement enclosure are obtained while eliminating extraneous information. An actual time period of 2 to 2-1/2 hr per sample is estimated for performing the analysis. However, because of grid transfer time, a total of 40 hr is necessary to approve or disapprove contractor clearance.

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TABLE I. DETECTION LIMITS

1.0	Filter Size	25 mm	37 mm
2.0	Effective Filtration Area	3.8 cm^2	8.55 cm^2
3.0	Average Area of Grid Opening (75 μm per side)	.00005625 cm^2	.00005625 cm^2
4.0	Number of Grid Openings	10	10
5.0	Sampling Volume--Detection Limit	3000--2.25 fibers/ liter	3000--5.07 fibers/ liter
6.0	Sampling Volume--Detection Limit	2000--3.38 fibers/ liter	2000--7.6 fibers/ liter
7.0	Sampling Volume--Detection Limit	1332--5.07 fibers/ liter	—

TEM ASBESTOS ABATEMENT ANALYSIS REPORT

Sample I.D. C701-275	Date Analyzed 1/16/86	Grid Box Misc. #23
IITRI I.D. C08820-C701	Accl. Voltage 100 kV	Grid Location B2 + B3
Date Sample Received 1/15/86	Beam Current 100 µA	Volume Sampled 2520 liters
Filter Type 25 mm Nuclepore	EM Magnification 20,000X	Method of Sampling Aggressive
Filtration Area (cm²) 3.8	Fluor. Screen Magnif. 1 µ = 16 mm	

Description of Location Northeast corner of enclosure Results 0.017 asbestos structures per cc

Grid Opening	Chrysotile and Amphibole Asbestos							Ambiguous	Non-Asbestos
	Fibers			Bundles		Clusters	Matrix		
	< 1 µ	1 to 5 µ	> 5 µ	< 5 µ	> 5 µ				
1 0 / 0									
2 / 0		1 1							
3 / 0					1				1
4 0 / 0									
5 / 15		1 1							
6 0 / 16		1					1	1	1
7 / 0		1							
8 / 0	1 1	1							
9 / 19	1	1		1				1	
10 / 0									1
TOTALS	3	8	0	1	1	0	1	2	3
Per Filter Area	9186	24,496	BDL*	3062	3062	BDL*	3062	6124	9186
No. per Liter	3.65	9.72	—	1.22	1.22	—	1.22	2.43	3.65
No. per cc	.004	.01	—	.001	.001	—	.001	.002	.004

*BDL = Below detection level

Detection Limit 1215 asbestos structures per m³

Area of G.O.--Start 63 x 63 @ 1000X - 64 x 64

Area of G.O.--End 63 x 63 @ 1000X - 61 x 62

20 (No. of G.O.s Examined) x .00006205 (Avg Area of G.O., cm²) = .001241 (Total Area Examined, cm²)

EM Description of Sample Medium loading; opaque spheres and combustion aerosol

Figure 1. An example of a completed data sheet.

APPENDIX E

NOTES ON THE USEPA PEER REVIEW ON ASBESTOS ANALYTICAL PROCEDURES, APRIL 24-25, 1986

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SUMMARY

Central to the meeting was discussion of the asbestos fiber contamination that MES found on the polycarbonate filter field blanks which were part of several large projects sponsored by EPA. In our review of the situation, we could find no evidence of in-laboratory contamination of the extent seen on the filters (10-20 asbestos structures per 10 grid openings). Our laboratory quality assurance blanks did not show levels approaching those found. I suggested that the contamination either existed in the polycarbonate filters beforehand (Nuclepore filter contamination is known to exist) or that fibers were carried into the preparation clean room on the filter cassettes and contaminated the filters during the carbon-coating step of the preparation.

PROCEDURAL NOTES

The sampling and preparation procedures, essentially described in Yamate, 1984 (1), are fairly straightforward and it is difficult to see how contamination could have occurred. The field blanks were reportedly brought into the work area but never opened. All project samples including field blanks were hand-carried to MES, logged in, and stored in a designated cabinet.

The samples were identified by PEI identification codes but there was no indication as to whether the filters were air samples or field blanks. Groups of cassettes were opened in the preparation room and the filters were coated with carbon in a vacuum evaporator without taking them out of the cassettes. TEM grids were prepared using a Jaffe wick apparatus. The grids were placed in clean grid boxes, transferred to the nearby microscope room, and analyzed on a transmission electron microscope.

RESULTS OF ANALYSIS

The samples were analyzed "in the blind" (without knowledge of their identification) according to the Yamate, 1984 procedures defining asbestos structures as fibers, bundles, clumps (clusters) or matrix particles. A summary of the asbestos counts (structures per 10 grid openings) and plots showing the size distribution of structures can be found elsewhere in these proceedings. Electron micrographs of a chrysotile structure and amphibole fiber found in one of the field blanks are shown in Figures 1 and 2. The filters were generally clear of other particulates. There was a considerable difference between the first sets of data and the last sets 9 and 10. In the first sets essentially no asbestos fibers were found by either the IITRI (Yamate) Laboratory or the McCrone Laboratory. In the last sets, the McCrone Laboratory found higher concentrations of asbestos fibers than PEI analysts working at the NIOSH lab or Pat Clark of EPA. When viewing one of the samples prepared by the PEI/NIOSH analysts in the EPA prepared by the PEI/NIOSH analysts in the EPA microscope, I saw a number of non-asbestos fibers which had not been seen in our preparations. Mr. Clark also remarked that these had not shown up in his preparations. They appeared to be formed of undissolved filter material which may have interfered with the PEI/NIOSH analysis. During the quick look we took at the EPA facility, Mr. Clark found 2 chrysotile fibers in one grid opening. This could be taken as an indication of the levels we reported (1-3 fibers per grid opening). A list of non-asbestos fibers McCrone found in the last sets of analyses is shown in Table I.

CONTAMINATION

When analyzing for ambient or near ambient levels of asbestos, it is important to take a range of precautions to prevent contamination. The following summarizes some of the precautions and quality assurance steps taken by McCrone Environmental Services, Inc.

FACILITY

1. No asbestos-containing materials were used in the construction of the facility.
2. The preparation room is isolated from the rest of the building.
3. The standard pressed-particle ceiling tiles have been replaced with tiles which do not contribute particles to the air.
4. All regular air conditioning ducts are blocked off.

5. Incoming air is forced through a HEPA filter before entering the room.

6. The HEPA unit supplies a positive pressure within the room forcing air out the door and out of the hood at all times.

7. All water used for cleaning or dilution is filtered through a reverse osmosis ultra filtration unit.

8. Tacky mats are used to collect any dirt from shoes.

QUALITY CONTROL

A number of QC blanks are run to check for contamination:

Filter only

Filter and Air

Filter and Water

Filter, Asher and Water

Drop Mount of Coated Grid (no filter)

In the past years, numerous QC blanks have been run. None have approached the 1 to 3 fibers per grid opening levels found in the field blanks. A plot of part of the QC blank control chart can be found in Figure 3. Although no laboratory blank filters were placed in the vacuum evaporator during carbon coating, no evidence of high contamination was seen in lab blanks run before and after the PEI/EPA filters were coated.

FILTER CONTAMINATION

At least some of the fibers found during the QC blank runs are probably contributed by the polycarbonate filters themselves.

Problems with contamination of polycarbonate filters have been reported by several researchers (2,3,4). The fiber contamination of the polycarbonate filters appears to vary from batch to batch and both chrysotile and amphibole fibers have been found. The levels of fiber contamination in several batches of polycarbonate filters are shown in Table 2.

QUALITY ASSURANCE

Some differences in laboratory results may be due to different procedures and some to the subjective judgement involved in the visual identification of asbestos fiber. McCrone runs duplicate analysis periodically and it is routine to have a second analyst analyze part of a sample as a check. Internal consistency within a lab can be maintained by checks and duplicate analyses but there is little to help in developing standard counting between laboratories. We had 5 analysts count the NBS Standard Reference Material (SMR-1876) and our results were very similar to those given on the SMR data sheet. We feel therefore that we count in a similar manner as the NBS analysts. However, our attempts to cross-check with two other laboratories have not been successful because the other labs have been too busy to do the samples. Recently we have become involved with an International Counting Trial out of Edinburgh, Scotland and look forward to an ASTM round robin which I understand is in the works.

THE POWERS RING

One interesting new idea for checking contamination of a filter uses a 37 mm base and a 25 mm cowl connected by a metal ring which covers part of the 37 mm filter. We field tested a prototype of the ring assembly (Figure 4). The industrial hygienist who conducted the field test, Mr. Kevin Brown of MES, made notes of the project and his comments can be found in the Appendix of this document. Several sections of the filter were analyzed and the results are given in Table 3. Unfortunately, the filter under the ring which should have been blank had some particulate material similar to that area not covered. It may be that a better seal is needed around the ring base. It would also seem that the base of the 37 mm cassette should be modified to prevent air from being pulled through the portion of the filter covered by the ring. In general, the device looks promising.

OTHER FILTERS

Partly because of the polycarbonate filter contamination problems, methods for collapsing membrane filters by chemical means have been developed to allow the advantages of membrane filter collection (low pressure drop, high filter efficiency, and high particle retention) to be combined with the analysis of the particles on a smooth, flat, featureless substrate. The collapsed membrane method described by Burdett and Rood (5) is based on earlier work in which the surface of a cellulose ester filter was first fused by exposure to a solvent vapor to form a smooth surface, and the filter was then coated and placed in a Jaffe washer (6,7). An important feature of the Burdett and Rood method is that the collapsed filter is etched in an oxygen plasma to expose fibers on the filter surface before coating. This step has been used with the preparation of filters for scanning electron microscopy (8). A similar method used by Dr. Daniel Crane at OSHA, Salt Lake City, substitutes the use of acetone for the more toxic solvent, dimethylformamide (DMF) used by Burdett and Rood, in the step of collapsing the filter and later in dissolving the filter away from the carbon film (9). A brief description of this unpublished method follows:

First, a wedge of the filter is placed on a glass cover slip, particle side up. This is then exposed to boiling acetone vapors to collapse the filter to about 20 micron thickness. This is done quickly to keep the filter from deforming and thereby changing the concentration of fibers. This process smooths out the surface of the filter and encapsulates some fibers in the top layer of the filter. The filter is then placed in a plasma etcher. The sample is etched sufficiently long to remove 7 to 10 microns of filter material. This exposes the fibers previously encapsulated. Carefully, the filter is removed from the asher. This process has the effect of leaving an extremely smooth surface on the filter so that no background roughness can obscure very small fibers. It is then coated with gold to firmly attach the particles on the surface of the filter. After being coated with gold, the filters are removed from the slides and a small (4 mm square) portion is placed particle side down on a TEM sample grid with a formvar or carbon substrate. This is then placed in a Jaffe wick device to dissolve the remaining membrane material leaving the fibers and other particulate on the grid. The samples are left in the Jaffe wicks overnight in a fume hood. Depending on conditions, the samples may be viewed sooner than this. When the filter material is gone, the grids are air dried and placed in the TEM for analysis.

CONCLUSIONS

Our first conclusion was that the contamination probably came in the filters themselves. A second possibility occurred to us during a quality circle meeting in which we reviewed the data. Paul Baron of NIOSH had reported that asbestos could become attached to the inside of the sampling cowl by static charge. It occurred to us that the fibers could be also on the outside of the cowl. The cassettes had been in an abatement project area and, during preparation for carbon coating, the fibers attached to the outside of the cassette might have contaminated the filters. To prevent this in the future, it will be our standard procedure to wash down incoming cassettes with a damp cloth and/or HEPA vacuum.

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Table I

Report on Non-Asbestos Fibers Found in Field Blank Samples

McCrone Environmental Services, Inc.

Sample ID	Lab ID	Non-Asbestos fibers/10 grid squares
W-1-B	1819-177	2.4 x .05 um Al Si
W-2-B	1819-178	3.0 x .05 um Si 1.2 x .05 um no EDS
W-3-B	1819-179	2.1 x .07 um no EDS 1.4 x .1 um Si
P-1-B	1819-180	1.2 x .1 um Ca Ci 2.1 x .05 Al Si
P-2-B	1819-181	1.5 x .05 um Si 1.0 x .05 um no EDS
P-3-B	1819-182	.5 x .05 um Si Al

Table II

Nuclepore Polycarbonate Filter Blank Analyses

Lot #	# Analyzed	Asbestos Structures/10 Grid Openings
83AV3B49	1	57
83B5A1	2	20-60
83D4A41	2	0-2
83D4AV42	2	0-4
83B9A36	2	1-2
83C4B72	3	2-3
83C4C48	17	0-5
83D4A36	7	0-4

Table III

Powers Ring Field Test

TEM Analysis Results

Grid #	Identification	Asbestos Structures/ 10 Grid Openings
1	Outside edge of filter	2
2	Underneath ring but near sampling area	6
3	In sampling area	6
4	Center of sampling area, center of filter	7

NOTE: All four grids showed particulate material, oil, gypsum, aluminum silicate fibers and sheet silicates.

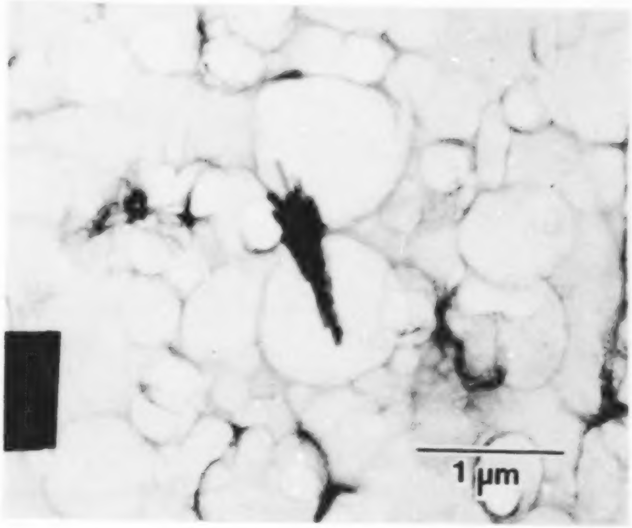
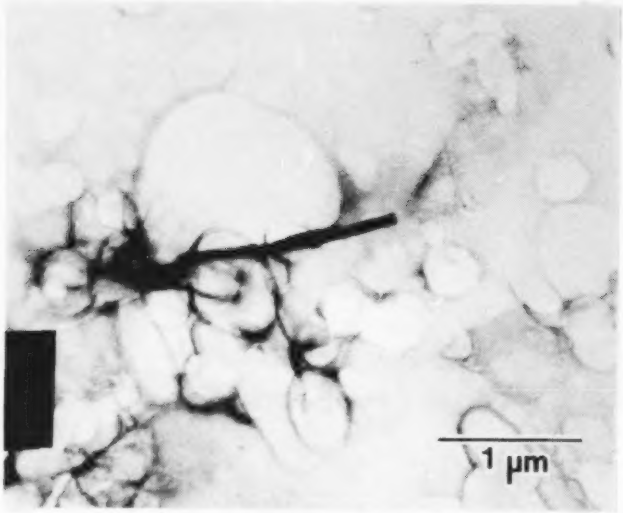


Figure 1 Chrysotile clump found in field blank ME-2373-2 [COR-QA-N-2]



[redacted]

Figure 2 Amphibole fiber found in field blank
ME-2373-2 [COR-QA-N-2]

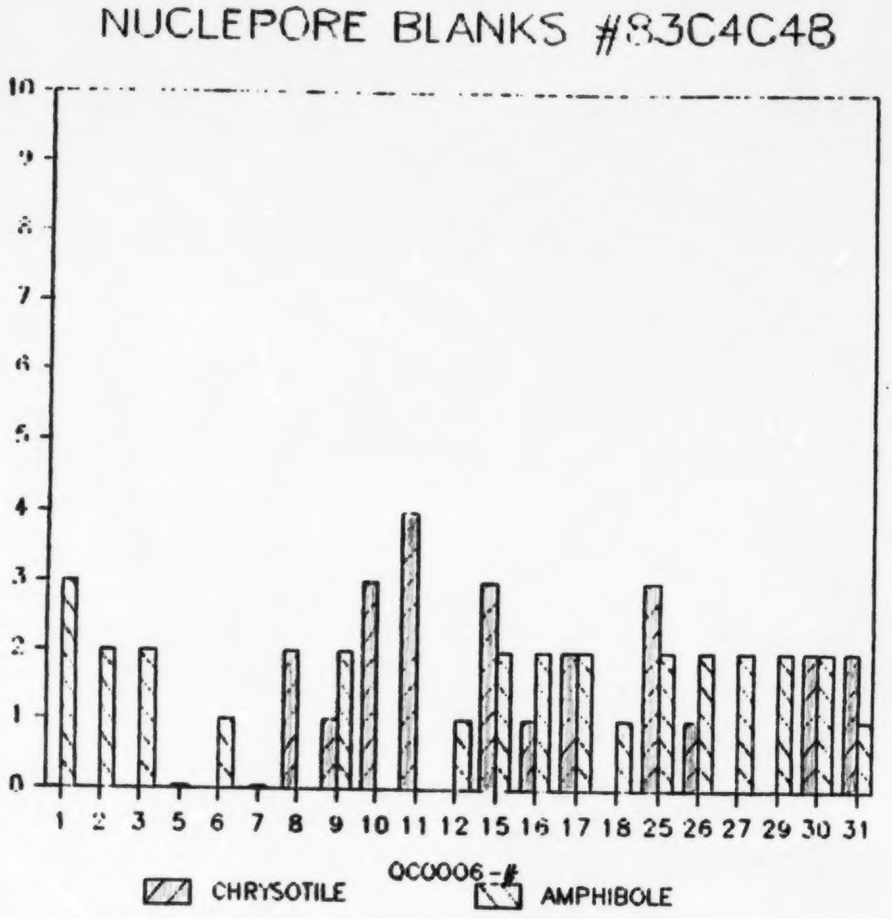


Figure 3 Part of Nuclepore Blank Control
Chart - y-axis = fibers per 10 grid openings
- x-axis = filter blank number

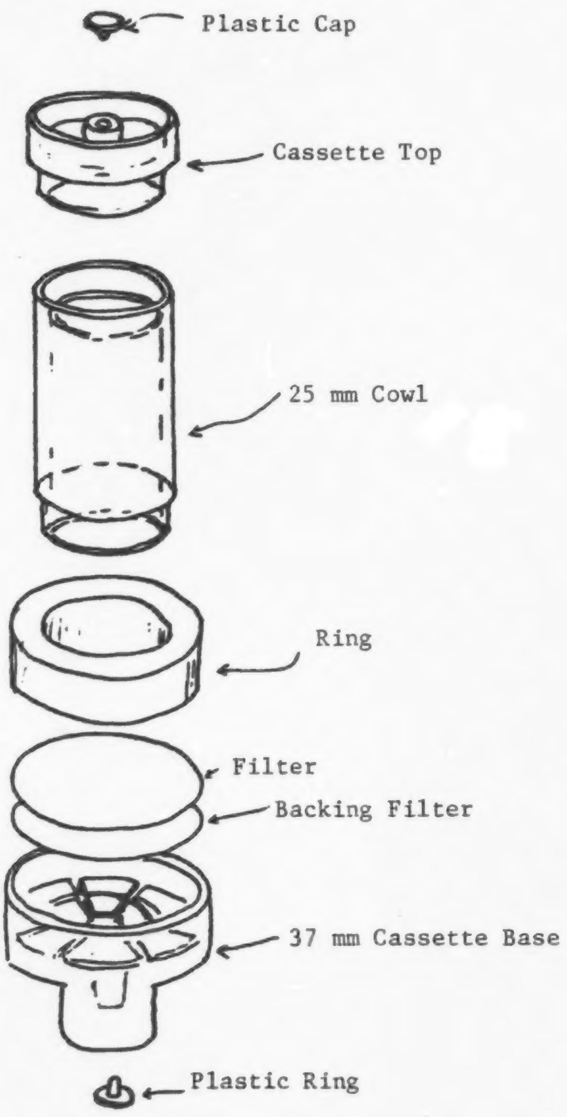

Plastic Cap
Cassette Top
25 mm Cowl
Ring
Filter
Backing Filter
37 mm Cassette Base
Plastic Ring

Figure 4 Powers Ring Assembly

Appendix

Observations of Field Use of the Powers Ring

Field testing of the Powers Ring on Thursday, February 27, 1986, at Georgia State University's Business Administration building proceeded without any problems. The filter assembly with the Powers Ring attached had no trouble fitting into the box for standard 37 mm cassettes. This allowed for easy transportation of the cassette to the sampling location.

The sample was setup in the first floor lobby area of the building. The possible problems arising from the increased weight of the cassette did not occur. The wood paneling in the lobby was smooth and clean enough that the duct tape used to secure the cassette held. Actually, the increased weight helped hold the lighter 25 mm cowl at the recommended tilted position.

The cowl presented no problems in the calibration of the sampling pump and the attaching of the base to the bottom of the cassette. There was a slight problem with trying to write on the cassette. The cowl limits the amount or area but writing smaller solved the problem. The Powers Ring did not increase the time it took to setup a sample nor was it noticeably louder or cause any unusual noises.

The sample was running and still in place when it was picked up approximately 22 hours later. No visible damage of leakage could be seen. The tape had held and the cassette was still in the required sampling position.

The purpose for using the Powers Ring is to allow the analyst to be able to use the same filter as a blank and a sample. This is done by using a 37 mm filter and fitting the ring over, thus cutting the effective filter area to a 25 mm filter.

There are several other advantages to using a ring.

1. The Ring reduces the effective filter area, thus reducing the time needed to achieve certain required detection limits.
2. The weight of the Ring enables the hygienist to position the filter and attached cowl in the necessary 45° position. The normal problem is that the smaller cowl (25 mm diameter) usually will rise over time because of its lack of weight.

The disadvantages of using the Powers Ring area as follows:

1. The Ring limits the amount of area the hygienist has to write on the cowl. This can be remedied by writing smaller or on another piece of paper;
2. The assumption must be made that the Ring's fit onto the filter is air tight. If it is not, the effective filter area would be inaccurate and thus, the corresponding fiber concentration data;
3. The increased weight of the filter assembly may cause samples to pull loose from the wall and fall. This is a problem only in areas where excessive amounts of tape cannot be used or in areas of high humidity.
4. Repeated use of the ring will require cleaning procedures to be adopted.

APPENDIX F

STUDIES OF ASBESTOS CONTAMINATION IN TEM ANALYSES

Richard Lee
Energy Technology Consultants
Monroeville, Pennsylvania 15146

This report summary presents the results of: 1) ETC'S quality control analysis of laboratory and field blanks, 2) comparison of TEM asbestos analysis using magnifications of 10000 and 19000, 3) Experiments to determine the asbestos contamination levels in Nuclepore™ and Millipore™ filters, and 4) ETC'S analysis of blank filters from the PEI study in which high blank valves were found by other laboratories. Each topic is summarized briefly.

LABORATORY AND FIELD BLANK ANALYSIS

ETC prepares and analyzes one blank laboratory filter with each group of samples prepared in a class 100 clean hood. The blank filter is carbon coated and prepared as the last sample in each group. Since we recommend that at least one field blank be submitted with each group of filters to be analyzed, we can compare the asbestos content of laboratory and field blanks. Table I contains the average and standard deviation for the two sets of blanks. The data indicated that field blanks average about 6 times higher than laboratory levels. ETC averaged approximately .5 fibers per laboratory blank filter. A complete report containing the fiber count sheet is attached.

What is the significance of the data? Contamination of field blank is a significant problem. The data suggests that the contamination in the field blank is not from the same source as that observed in the ETC laboratory. The contamination may introduce at anytime from loading of the cassettes to removal of the filter from the cassette.

10000 VS 19000 ANALYSES

The sample methods developed for EPA recommended that TEM asbestos analysis be performed at 19000 times magnification. There appeared to be very little or no data on whether 20K is optimal. Several different approaches have been evaluated by ETC. They include:

1. A 2000X nominal magnification with the operator viewing the field through a 10X binocular

   This technique has promise for low magnification screening of samples.
2. 10000 X analysis
3. 19000 X analysis

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ETC analyzed 150 areas at 10000X and 19000X as shown in Table II. Six operators were involved. Figure 1 contains a summery of the 10000X vs 19000X analysis. In essence no significant difference in the results were observed. We have qualitatively observed that the 19000 magnification is better for higher fiber loading and the 10K magnification better for densely loaded air samples with a large number of structures associated with the matrix material. In both cases the preferred way minimizes the probability of double counting, and/or missing structures through improper repositioning after the fiber identification process. We also have noted that the largest source of variability in fiber identification at either magnification may occur for fibers <.4 µm in length and would strongly recommend that a minimum length criteria be adopted.

In summery, we believe that 10000 and 19000 analyses both have application and that the laboratory should choose the one which best suits the sample.

ASBESTOS CONTAMINATION OF MILLIPORE™ AND NUCLEPORE™ FILTER

Reports of high contamination level in blank samples have led to concern that the filter media may be contaminated. ETC has experienced minor levels of contamination but nothing approaching the levels reported elsewhere (15-60 fibers per analysis). ETC undertook a small study designed to determine whether the Nuclepore™ or Millipore™ filter contained asbestos in the bulk of the filter. The results of the ongoing QA/QC asbestos analysis described earlier suggest that surface contamination of the filter was unlikely.

ETC prepared groups of 0, 1, 5, and 10 Nuclepore™ filters for low temperature ashing. The ashed residues were suspended in acetone and filtered onto .2µm Nuclepore™ filters. The samples were then analyzed by TEM. The results, illustrated in Table III and Figure 2, clearly demonstrate an increase in the number of fibers detected as the number of fibers analyzed was increased. The results of the individual analyses are attached. The majority (2/3) of the fibers detected were amphibole, and 1/3 were chrysotile.

The experiment was repeated using 37mm .2µm Millipore™. Again asbestos contamination was confirmed. The results for the 10 filter ashing were lower than expected; however, this situation was corrected when the filters were finely divided before ashing. The results of the Millipore™ experiments are shown in Table IV and Figure 3. In their case the predominant fiber type was chrysotile.

In subsequent conversations with Millipore Corporation, it was indicated that the .2μm filter had no special precaution taken to eliminate asbestos contamination. Millipore suggested that the experiment be repeated on the .8 μm or 1.2μm filters recommended for air sampling where they do take special precautions to eliminate asbestos contamination in the manufacture of these filters. ETC has received their filters, but the experiments are not completed.

In summary, the experiments demonstrate that bulk contamination of the filter media occur, and can pose a problem if the filters are ashed. Use of the Modified Jaffee Wick technique should minimize the observed levels although occasionally some fibers must intercept the filter surface and become part of the TEM sample.

ETC would recommend that field filters be prescreened until it is established that the bulk contamination has been minimized. If at all possible, ashing of filter samples not specially manufactured to eliminate bulk contamination should be avoided.

ETC Analysis of PEI Blank Samples

ETC requested and received 6 blank analyzed by Pat Clark, NSEPA Cincinnati. Significant contamination had been reported for their filter (see Table V). ETC's data are attached and included in Table VI. In summary, contamination was observed, but at a much lower level than reported by McCrone or PEI. ETC's results agreed generally with EPA's analysis.

One observation was made by ETC personnel. The carbon films were extremely thick, leading to poor clearing of the filter and making fiber identification difficult. This could have contributed to the unidentified PEI fibers.

The original high asbestos counts obtained by McCrone are not explicable in terms of the ETC and EPA results. It would appear that McCrone filters were separately contaminated, or possible artifacts were introduced during sample preparation. In any event, the McCrone grids should be reanalyzed, or additional samples prepared from their filters and analyzed.

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TABLE I
COMPARISON OF BLANK LEVEL CONCENTRATIONS

As of May 15, 1986

Based on equivalent volumes for
unit fiber concentration
(treating 0 count as 1)

ETC Q/A Blank 46 Samples	Client Blank 54 Samples
$\bar{x} = 0.007$ f/cc	$\bar{x} = 0.026$ f/cc
$s = 0.004$	$s = 0.030$

Based on equivalent volumes for
unit fiber concentration
(treating 0 count as 0)

ETC Q/A Blank 46 Samples	Client Blank 54 Samples
$\bar{x} = 0.004$ f/cc	$\bar{x} = 0.025$ f/cc
$s = 0.006$	$s = 0.031$

Equivalent volumes necessary for 0.005 f/cc
background level

25mm = 1500 liters
37mm = 3000 liters
47mm = 4000 liters

TABLE II

Comparison of 10000X and 19000X Counts
(150 areas analyzed)

10000X	19000X
$\overline{X}$ = .38 fibers per area	$\overline{X}$ = .44 fiber per area
σ = 1.55	σ = 1.77

125 of 150 areas had identical counts

17 of 150 areas differed by 1 fiber

5 of 150 areas differed by 2 fibers

3 of 150 areas differed by 3 fibers

19000X had the highest count 13 times

10000X had the highest count 12 times

TABLE III

Nuclepore 0.2 µm

No. Filters Low Temp. Ashed LTA	Lot#	Length* >1 micron	Length* < 1 micron	Total	Amph.	Chry
1	83C4B66	8	5	13	8	5
1	83C4B66	9	6	15	8	7
5	83C4B66	6	15	21	12	9
10	83C4B66	14	6	20	13	7
10	83C4B66	4	7	11	7	4
1	83D4C11	5	11	16	10	6

* Total counts are not based on 10 analyzed fields by TEM
See graphs with concentrations vs # filter (LTA)

TABLE IV

Millipore Type GS 0.22 μm

No. Filters Low Temp. Ashed LTA	LOT#	------------Length----------* >1 micron	< 1 micron
1	HOD62232C	5	4
1	HOD62232C	1	9
3	HOD62232C	4	7
10	HOD62232C	2	3
10	HOD62232C	11	13
1	HDS641635	5	2
2	HDS641635	3	9
5	HDS641635	10	20
10	HDS641635	4	3

* Total counts are not based on 10 analyzed fields by TEM
See graphs with concentrations vs # filter (LTA)

TABLE V(i)

TABLE 1 (continued)

Sample group	Date	Study	Sample No.	Location of carbon coating	McCrone Structures/10 grid openings, 20,000X			NIOSH /PEI Structures/10 grid openings, 20,000X			Yamate Structures/20 grid openings 20,000X			ETC Structures/10 grid openings 20,000X *			EPA NIOSH PREPARED Structures/10 grid openings 20,000X			EPA EPA PREPARED Strucutres/10 grid openings 20,000X		
					As[a]	Nonas[b]	Amb[c]	As	NOID[d]	Amb	As	Nonas	Amb	As	Nonas	Amb	As	Nonas	Amb	As	Nonas	Amb
6	7/31 to 8/4/85	Columbus East - Mike Beard's QA Samples	E-CE-N-1174	NIOSH										[g]	[g]							
			E-CE-N-1175	NIOSH										0	0							
			E-CE-N-1176	NIOSH										3	1							
			E-CE-N-1177	NIOSH										5	9							
			E-CE-N-1204	NIOSH										1	1							
			E-CE-N-1205	NIOSH										25	6							
			E-CE-N-1206	NIOSH										0	8							
			E-CE-N-1207	NIOSH										1	0							
7	8/16 to 8/20/85	Houston Post-abatement	E-HI-N-1263	McCrone	3	0	0															
			E-HI-N-1271	McCrone	22	0	1															
			E-HI-N-1277	McCrone	42	0	0	5	11	6												
			E-HI-N-1287	McCrone	33	1	0															
8	11/18 to 11/22/85	Columbus East Postabatement, Phase 2 (OTS)	E-CE-N-1350	NIOSH																		
			E-CE-N-1351	NIOSH																		
9	2/86	Samples prepared by Bill Cain, EPA	W-1-A,B[h]	A(NIOSH)/ B(McCrone)[i]	22	0	0	0	21	1				5	0		5	18	2	1	12	6
			W-2-A,B[h]		33	0	0	0	9	3				2	0		3	22	4	7	11	2
			W-3-A,B[h]		17	0	0	2	8	1				8	0		-	-	-	-	-	-
10	2/86	Samples prepared by Ann Crone, PEI	P-1-A,B[h]	A(NIOSH)/ B(McCrone)[i]	20	0	0	0	16	0				10	0		5[j]	16[j]	2[j]	0	10	0
			P-2-A,B[h]		16	0	0	0	16	2				3	2		0	32	1	7	14	1
			P-3-A,B[h]		19	0	0	0	14	2				4	0		-	-	-	-	-	-

[a] As = asbestos structures. Includes chrysotile and amphibole fibers, bundles, clumps, and matrices.

[b] Nonas = nonasbestos structures.

[c] Amb = ambiguous structures.

[d] NOID = no identification of structure.

[e] More than 100 grid openings were examined at 10,000X. Number shown indicates an estimate of the number of structures counted in 10 grid openings, assuming all structures to be asbestos. Breakdown on structure composition was not given.

[f] 95 grid openings were examined at 20,000X. Number shown indicates an estimate of the number of structures counted in 10 grid openings.

[g] Sample damaged by carbon coating.

[h] Sample filters were prepared and divided into halves. One portion of the filter was labelled "A" and was analyzed by NIOSH. The remaining portion of the filter was labelled "B" and was analyzed by McCrone.

[i] The filter halves labelled "A" were carbon coated by NIOSH. The filter halves labelled "B" were carbon coated by McCrone.

[j] Only seven grid openings were examined due to equipment breakdown.

(i) Table V was provided through the courtesy of PEI associates.

* 10000X Magnification

TABLE VI.

SAMPLE ETC REPORT

May 29, 1986

Ms. Ann Crone
PEI Associates
11499 Chester Road
Cincinnati, OH 45246

RE: TEM Asbestos Results for Samples T-3339-T3344R
ETC Job No. 8604131

Dear Ms. Crone:

Enclosed are the results from the scanning transmission electron microscopy (TEM) asbestos analysis of the above referenced samples. The samples were collected by RTI personnel on 37 mm polycarbonate filters. Sample preparation for the TEM analysis involved directly coating the filter with a thin layer (~400Å) of carbon by evaporative deposition under vacuum. The samples were cleared with chloroform, leaving the particles attached to the carbon film. Several grids containing approximately 0.3 mm sq. of filter were prepared for each samples. The samples were analyzed at a magnification of 10000X and 19000X.

Enclosed is a brief summary of the results of the analysis for the samples. Table I list the sample identification numbers, sample volumes, and areas analyzed. The actual counts and estimated ranges for asbestos and nonasbestos fibers are summarized in Table II.

The estimated counts shown on Table II are based on Poisson distrubution. Classifications are derived from the following:

1. NONDETECTABLE - Fewer than five fibers detected. Assuming a blank count corresponding to one fiber and Poisson statistics ($a = 0.05$), this implies that the result is indistinguishable from background.

2. INDETERMINATE - Between 5 and 11 fibers detected. In this range, the count is distinguishable from background but the probability that a repetition of the analysis could yield a results indistinguishable from background is greater than 5%.

3. DETECTABLE - More than 11 fibers detected. The probability that a repetition of the analysis would be indistinguishable from background is less than 5%.

4. MEASUREABLE - More than 100 fibers detected. This specifies that uncertainty is less than 10%.

The uncertainties specified above comprehend only counting statistics. Additional uncertainties resulting from other factors such as sample collection and preparation are not included.

Table III presents the original filter concentration (f/cm^2) for all asbestos and nonasbestos fibers detected, while Table IV summarizes the ambient air concentrations (f/cc).

Copies of the fiber counts are presented in Appendix A. These data sheets present results for fibers that had a length-to-width ratio of $\geq$ 3:1. Each fiber is identified by field and fiber identification numbers; also recorded are length and width measurements in micrometers and fiber chemistry.

Appendix B contains sample sheets and graphs comparing the results of analyses using magnifications of 10000X and 19000X

If you have any questions, feel free to call Gary Casuccio or me.

Very truly yours,

George R. Dunmyre
Manager - Laboratory Services

Enc.
GRD/jrr

TABLE I
Summary of Pertinent Information
Used to Calculate the Concentration of Asbestos

Sample Identification		Volume	Area Analyzed
ETC#	Client#	liters	mm^2
T3339	ME-1819-177-W-1-B	Blank	0.0550
T3339R	ME-1819-177-W-1-B	Blank	0.0550
T3340	ME-1819-178W-2-B	Blank	0.0550
T3340-2	ME-1819-178-W-2-B	Blank	0.0550
T3340R	ME-1819-178-W-2-B	Blank	0.0550
T3341	ME-1819-179-W-3-B	Blank	0.0550
T3341R	ME-1819-179-W-3-B	Blank	0.0550
T3342	ME-1819-180-P-1-B	Blank	0.0550
T3342R	ME-1819-180-P-1-B	Blank	0.0550
T3343	ME-1819-181-P-2-B	Blank	0.0550
T3343R	ME-1819-181-P-2-B	Blank	0.0550
T3344	ME-1819-182-P-3-B	Blank	0.0550
T3344R	ME-1819-182-P-3-B	Blank	0.0550

TABLE II

Summary of TEM Fiber Counts

Sample Identification		Asbestos			Nonasbestos		
ETC#	Client#	Actual	Estimated	Class	Actual	Estimated	Class
T3339 §	ME-1819-177-W-1-B	5	2- 12	Indeterminate	0	0- 4	Nondetectable
T3339R §	ME-1819-177-W-1-B	6	2- 13	Indeterminate	1	0- 6	Nondetectable
T3340 §	ME-1819-178W-2-B	2	0- 7	Nondetectable	0	0- 4	Nondetectable
T3340-2 §	ME-1819-178-W-2-B	0	0- 4	Nondetectable	0	0- 4	Nondetectable
T3340R §	ME-1819-178-W-2-B	3	0- 9	Nondetectable	1	0- 6	Nondetectable
T3341 §	ME-1819-179-W-3-B	8	3- 16	Indeterminate	0	0- 4	Nondetectable
T3341R §	ME-1819-179-W-3-B	15	8- 25	Detectable	5	2- 12	Indeterminate
T3342 §	ME-1819-180-P-1-B	10	5- 18	Indeterminate	0	0- 4	Nondetectable
T3342R §	ME-1819-180-P-1-B	15	8- 25	Detectable	0	0- 4	Nondetectable
T3343 §	ME-1819-181-P-2-B	3	0- 9	Nondetectable	2	0- 7	Nondetectable
T3343R §	ME-1819-181-P-2-B	0	0- 4	Nondetectable	0	0- 4	Nondetectable
T3344 §	ME-1819-182-P-3-B	4	1- 10	Nondetectable	0	0- 4	Nondetectable
T3344R §	ME-1819-182-P-3-B	6	2- 13	Indeterminate	0	0- 4	Nondetectable

§ Blank

TABLE III

Original Filter Concentration (fibers per cm^2) Observed During the Analysis

Sample Identification		----Asbestos (x10^3)-----		---Nonasbestos (x10^3)---	
ETC#	Client#	Actual	Estimated	Actual	Estimated
T3339	§ ME-1819-177-W-1-B	9.1	3.6-21.8	0.0	0.0-7.3
T3339R	§ ME-1819-177-W-1-B	10.9	3.6-23.6	1.8	0.0-10.9
T3340	§ ME-1819-178W-2-B	3.6	0.0-12.7	0.0	0.0-7.3
T3340-2	§ ME-1819-178-W-2-B	0.0	0.0-7.3	0.0	0.0-7.3
T3340R	§ ME-1819-178-W-2-B	5.5	0.0-16.4	1.8	0.0-10.9
T3341	§ ME-1819-179-W-3-B	14.5	5.5-29.1	0.0	0.0-7.3
T3341R	§ ME-1819-179-W-3-B	27.3	14.5-45.5	9.1	3.6-21.8
T3342	§ ME-1819-180-P-1-B	18.2	9.1-32.7	0.0	0.0-7.3
T3342R	§ ME-1819-180-P-1-B	27.3	14.5-45.5	0.0	0.0-7.3
T3343	§ ME-1819-181-P-2-B	5.5	0.0-16.4	3.6	0.0-12.7
T3343R	§ ME-1819-181-P-2-B	0.0	0.0-7.3	0.0	0.0-7.3
T3344	§ ME-1819-182-P-3-B	7.3	1.8-18.2	0.0	0.0-7.3
T3344R	§ ME-1819-182-P-3-B	10.9	3.6-23.6	0.0	0.0-7.3

§ Blank

TABLE IV

Ambient Fiber Concentrations (fibers per cc)
Observed During the TEM Analysis

Sample Identification		---------Asbestos--------		-------Nonasbestos-------	
ETC#	Client#	Actual	Estimated	Actual	Estimated
T3339	§ ME-1819-177-W-1-B	0.026	0.01- 0.06	<0.005*	0.00- 0.02
T3339R	§ ME-1819-177-W-1-B	0.031	0.01- 0.07	0.005	0.00- 0.03
T3340	§ ME-1819-178W-2-B	0.010	0.00- 0.04	<0.005*	0.00- 0.02
T3340-2	§ ME-1819-178-W-2-B	<0.005*	0.00- 0.02	<0.005*	0.00- 0.02
T3340R	§ ME-1819-178-W-2-B	0.016	0.00- 0.05	0.005	0.00- 0.03
T3341	§ ME-1819-179-W-3-B	0.041	0.02- 0.08	<0.005*	0.00- 0.02
T3341R	§ ME-1819-179-W-3-B	0.078	0.04- 0.13	0.026	0.01- 0.06
T3342	§ ME-1819-180-P-1-B	0.052	0.03- 0.09	<0.005*	0.00- 0.02
T3342R	§ ME-1819-180-P-1-B	0.078	0.04- 0.13	<0.005*	0.00- 0.02
T3343	§ ME-1819-181-P-2-B	0.016	0.00- 0.05	0.010	0.00- 0.04
T3343R	§ ME-1819-181-P-2-B	<0.005*	0.00- 0.02	<0.005*	0.00- 0.02
T3344	§ ME-1819-182-P-3-B	0.021	0.01- 0.05	<0.005*	0.00- 0.02
T3344R	§ ME-1819-182-P-3-B	0.031	0.01- 0.07	<0.005*	0.00- 0.02

* Below unit fiber concentration
§ Calculations for 37 mm blanks based upon 3000 liters

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3344R
Client Sample #: ME-1819-182-P-3

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	No fibers								
5	1	0.80	0.10	Amphibole			√		
6	1	1.60	0.10	Amphibole			3497		
7	1	1.10	0.15	Chrysotile	Bundle >6		√		
	2	1.00	0.15	Amphibole			√		
8	1	1.10	0.30	Chrysotile	Bundle >6		√		
	2	0.60	0.15	Amphibole			√		
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 6

Total Asbestos: 6
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3344
Client Sample #: ME-1819-182-P-3

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	No fibers								
5	1	0.70	0.15	Amphibole			3471		
6	No fibers								
7	1	1.00	0.15	Chrysotile	Bundle 6		√		Tight.
	2	1.10	0.20	Amphibole			3472	3473	
8	1	1.00	0.15	Chrysotile	Bundle 5		√	3499	Tight
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 4

Total Asbestos: 4
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3343R
Client Sample #: ME-1819-181-P-2

Volume: Blank
Microscope: 100 CX
Magnification: 19000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	No fibers								
5	No fibers								
6	No fibers								
7	No fibers								
8	No fibers								
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 0

Total Asbestos: 0
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3339R
Client Sample #: ME-1819-177-W-1

Volume: Blank
Microscope: 100 CX
Magnification: 19000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	2.00	0.40	Amphibole			√		
2	No fibers								
3	1	1.40	0.10	Chrysotile	Bundle >10		√		
	2	1.00	0.25	Amphibole			√		
4	1	0.40	0.10	Nonasbestos					
5	No fibers								
6	1	0.50	0.05	Chrysotile	Cluster 4				
	2	0.60	0.05	Chrysotile	Cluster >5		√		
7	1	0.60	0.10	Chrysotile	Bundle >6				
8	No fibers								
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 7

Total Asbestos: 6
Total Nonasbestos: 1

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3339
Client Sample #: ME-1819-177-W-1

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	2.00	0.20	Amphibole			3468		
2	No fibers								
3	1	1.40	0.10	Chrysotile	Bundle >10		√		
	2	1.00	0.50	Amphibole			√		
4	No fibers								
5	No fibers								
6	No fibers								
7	1	0.60	0.10	Chrysotile	Bundle >6		√		
8	No fibers								
9	1	0.60	0.05	Chrysotile					
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 5

Total Asbestos: 5
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3340
Client Sample #: ME-1819-178W-2-

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	No fibers								
5	No fibers								
6	1	3.00	0.15	Amphibole			3469		
7	No fibers								
8	No fibers								
9	1	0.50	0.02	Chrysotile			√		
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 2

Total Asbestos: 2
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3340-2
Client Sample #: ME-1819-178-W-2

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	No fibers								
5	No fibers								
6	No fibers								
7	No fibers								
8	No fibers								
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 0

Total Asbestos: 0
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3340R
Client Sample #: ME-1819-178-W-2

Volume: Blank
Microscope: 100 CX
Magnification: 19000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	0.60	0.15	Chrysotile	Bundle		√		with matrix
2	1	0.80	0.10	Chrysotile			√		Touching next fiber
	2	0.10	0.02	Chrysotile					Touching.
3	No fibers								
4	No fibers								
5	No fibers								
6	No fibers								
7	No fibers								
8	1	0.50	0.05	Unknown					
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 4

Total Asbestos: 3
Total Nonasbestos: 1

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3341
Client Sample #: ME-1819-179-W-3

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	No fibers								
4	1	0.60	0.05	Chrysotile		√			
5	No fibers								
6	1	1.60	0.10	Amphibole			3475		
	2	0.90	0.05	Chrysotile					
	3	0.80	0.05	Chrysotile					
	4	0.60	0.15	Chrysotile	Bundle 6				
7	No fibers								
8	1	0.90	0.10	Amphibole			√		
	2	2.50	0.20	Amphibole			3477		
9	No fibers								
10	1	1.00	0.05	Chrysotile			√		

Total Fields Counted: 10
Total Fibers Counted: 8

Total Asbestos: 8
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3341R
Client Sample #: ME-1819-179-W-3

Volume: Blank
Microscope: 100 CX
Magnification: 19000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	0.50	0.05	Amphibole				X	
2	No fibers								
3	No fibers								
4	1	0.50	0.05	Nonasbestos					
	2	0.50	0.05	Nonasbestos					
5	1	0.60	0.05	Chrysotile					
	2	0.50	0.10						
	3	0.90	0.20	Talc			3478		?
6	1	1.50	0.10	Amphibole				3475	
	2	0.70	0.05	Chrysotile					
	3	0.60	0.05	Chrysotile			√		
	4	0.60	0.15	Chrysotile	Bundle >4		√		
	5	0.30	0.05	Chrysotile					Touching
7	1	10.50	0.15	Nonasbestos					
	2	1.00	0.10	Amphibole			3479		
	3	0.60	0.05	Chrysotile	Cluster 6				
8	1	1.00	0.10	Amphibole					
	2	2.50	0.20	Amphibole				3476	See photo 3476 & 3477
	3	0.60	0.05	Chrysotile	Cluster >7		√		
	4	3.20	0.20	Amphibole					
9	1	1.20	0.10	Chrysotile	Bundle >3		√		
	2	2.20	0.20	Amphibole					
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 20

Total Asbestos: 15
Total Nonasbestos: 5

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3343
Client Sample #: ME-1819-181-P-2

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	No fibers								
2	No fibers								
3	1	0.40	0.05	Chrysotile	Cluster >5				Coated; CHR in matrix.
	2	1.00	0.10	Unknown	Matrix				
4	1	0.30	0.05	Amphibole			3498		
5	No fibers								
6	No fibers								
7	1	0.35	0.05	Chrysotile					
	2	0.20	0.05	Unknown					
8	No fibers								
9	No fibers								
10	No fibers								

Total Fields Counted: 10
Total Fibers Counted: 5

Total Asbestos: 3
Total Nonasbestos: 2

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3342R
Client Sample #: ME-1819-180-P-1

Volume: Blank
Microscope: 100 CX
Magnification: 19000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	0.50	0.10	Chrysotile					
2	1	0.30	0.05	Chrysotile					
	2	2.20	0.10	Chrysotile					
3	No fibers								
4	1	1.00	0.15	Amphibole					
5	1	0.30	0.05	Chrysotile					
	2	0.30	0.05	Chrysotile					touching previous fiber
6	No fibers								
7	1	0.50	0.10	Chrysotile					
8	1	0.35	0.05	Chrysotile					
9	1	1.30	0.15	Chrysotile	Bundle 5		√		tight
	2	1.00	0.05	Chrysotile			√		
	3	0.60	0.05	Chrysotile			√		
	4	0.50	0.10	Chrysotile					
10	1	0.75	0.10	Amphibole			√		
	2	0.40	0.10	Chrysotile	Cluster 8				
	3	0.25	0.05	Chrysotile	Cluster 4				

Total Fields Counted: 10
Total Fibers Counted: 15

Total Asbestos: 15
Total Nonasbestos: 0

Client name: RTI/EPA
ETC Job #: 8604131
ETC Sample # T3342
Client Sample #: ME-1819-180-P-1

Volume: Blank
Microscope: 100 CX
Magnification: 10000X
Filter: 37 mm

Field	Fiber	Length	Width	Fiber Type	Morphology	EDS	SAED	Photo	Comment
1	1	0.50	0.02	Chrysotile			√		
2	No fibers								
3	1	0.50	0.05	Chrysotile					
4	No fibers								
5	No fibers								
6	1	1.20	0.15	Chrysotile	Bundle >3		√		
7	No fibers								
8	No fibers								
9	1	1.50	0.15	Chrysotile	Bundle >3		√		
	2	0.60	0.05	Chrysotile	Cluster 8		√		
	3	1.10	0.05	Chrysotile			√		
10	1	0.80	0.10	Amphibole	Bundle 3		3470		
	2	0.60	0.05	Chrysotile	Cluster 8				
	3	0.90	0.05	Chrysotile					
	4	0.60	0.05	Chrysotile			√		

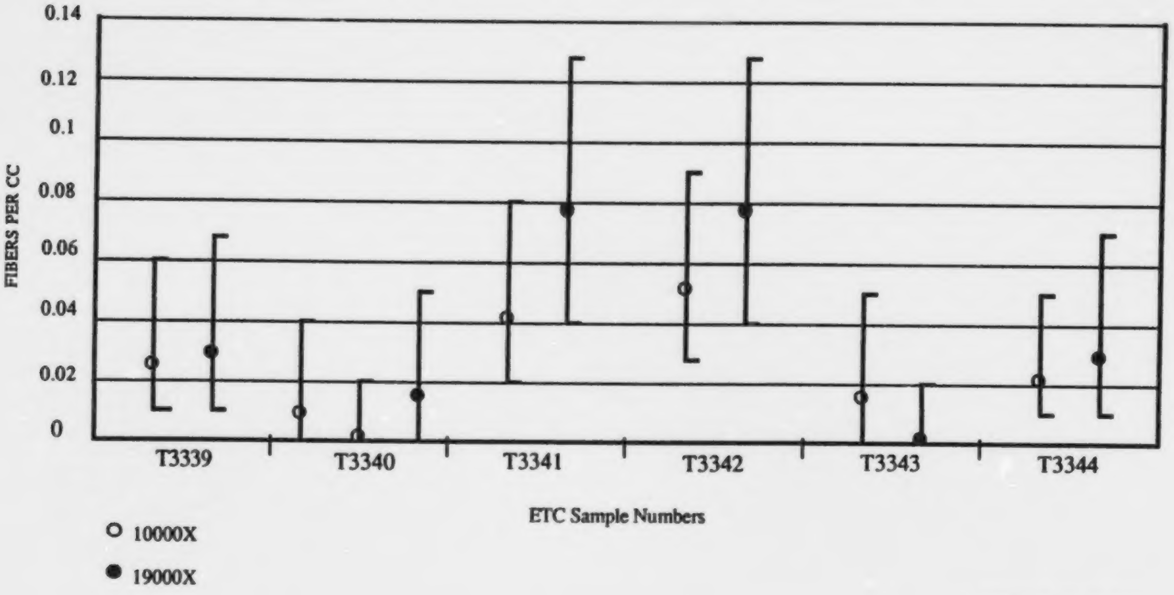
Total Fields Counted:	10
Total Fibers Counted:	10

Total Asbestos:	10
Total Nonasbestos:	0

FIGURE 1

Asbestos Fiber Concentrations (fiber per cc)
10000X vs. 19000X

Upper and Lower Limits Based on Poisson Distribution



ETC Job Number 86-04131

FIGURE 2

ASHING OF NUCLEPORE POLYCARBONATE FILTERS FOR ASBESTOS FIBERS

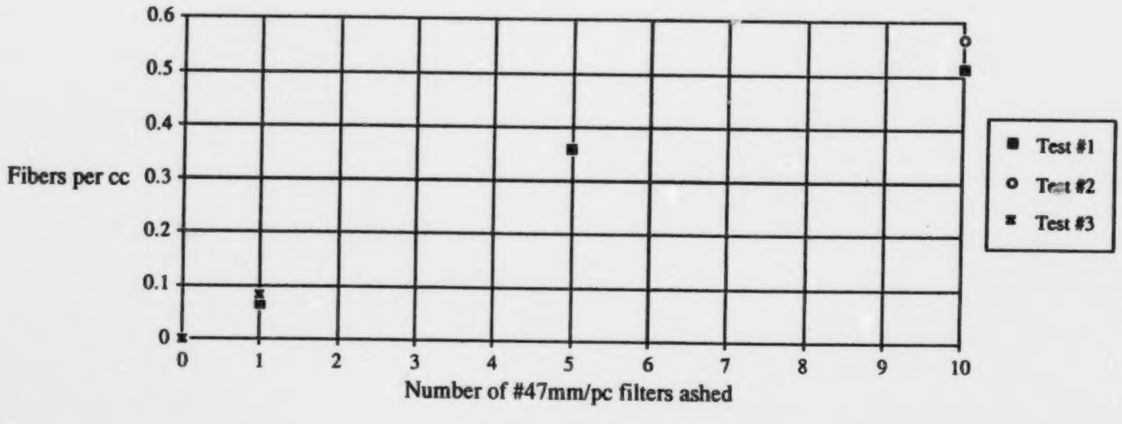
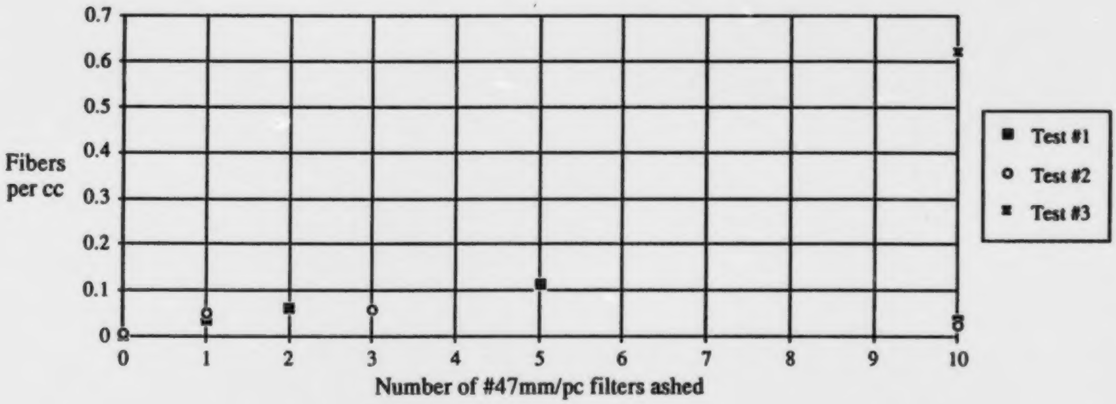


FIGURE 3

ASHING OF MILLIPORE CELLULOSE ESTER FILTERS FOR ASBESTOS FIBERS



APPENDIX G

THE 7402 METHOD FOR TEM

James W. Carter
National Institute for Occupational
Safety and Health
Cincinnati, Ohio 45226

I am sending you a copy of the revised method 7402 which is the first printing of a NIOSH TEM method. A summary of my opinions about TEM methods is as follows:

1. Mixed cellulose ester (MCE) filters (0.8 µm pore size) are acceptable media for TEM analysis. When prepared properly, the samples have a clean, even background free from spurious filter residue. The only noticeable exceptions which I have encountered over the years are filters which are overloaded, and filters which contain a lot of water and are not desiccated before clearing. When MCE filters are prepared by the NIOSH technique described in the accompanying paper there is little, if any, particle or fiber loss.

2. As Eric Steel has noted, there can be vast differences between instruments depending upon age and quality of upkeep. I would recommend that an instrument check be included in the method to determine the quality of the image and stage controls.

3. The nomenclature for identifying asbestos fibers should be standardized so that there is no question about what is meant by ambiguous, unidentified, or non-asbestos fibers.

We have found with the Phase Contrast Method that it is nearly impossible to get two different laboratories to perform a microscope method exactly the same way. There is a tendency for labs to pick the things they like and to ignore the things they do not like, especially if it involves purchasing something new. Of course, the force of law or even the illusion of it has a way of standardizing procedures.

I think you have a good start with Yamate's extensive background work and the advice from Eric Steel and Steve Hayward at the meeting. Do not hesitate to call if you have further questions.

Sincerely yours,

James W. Carter

James W. Carter
Physical Scientist
Monitoring Research Section
Monitoring and Control Research Branch
Division of Physical Sciences
and Engineering

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FORMULA: various

M.W.: various

ASBESTOS FIBERS

METHOD: 7402
ISSUED: 5/15/86

OSHA: 2.0 asbestos fibers (>5 µm long/mL)
NIOSH: 0.1 asbestos f/mL [1]
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

PROPERTIES: solid, fibrous

SYNONYMS: actinolite asbestos [CAS #13768-00-8], grunerite asbestos (amosite) [CAS #12172-73-5], anthophyllite asbestos [CAS #17068-78-9], chrysotile asbestos [CAS #12001-29-5], crocidolite asbestos [CAS #12001-28-4], tremolite asbestos[CAS #14567-73-8].

SAMPLING

SAMPLER: FILTER (0.8 to 1.2 µm cellulose ester membrane, 25-mm diameter; conductive cassette)

FLOW RATE*: 0.5 to 16 L/min (see step 5)

VOL-MIN*: 400 L @ 0.1 fiber/mL
-MAX*: 2000 L
*Adjust to give 100 to 1300 fibers/mm^2

SHIPMENT: in antistatic packing material

SAMPLE STABILITY: stable

FIELD BLANKS: 10% ($\geq$2) of samples

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: not determined

OVERALL PRECISION (s_r): see EVALUATION OF METHOD

MEASUREMENT

TECHNIQUE: MICROSCOPY, TRANSMISSION ELECTRON (TEM)

ANALYTE: asbestos fibers

SAMPLE PREPARATION: modified Jaffe wick

EQUIPMENT: transmission electron microscope; energy dispersive X-ray detector and analyzer

CALIBRATION: qualitative SAED/EDX; calibration of TEM and EDS system magnification

RANGE: 100 to 1300 fibers/mm^2 filter area [6]

ESTIMATED LOD: 1 confirmed asbestos fiber above 95% of expected mean blank value

PRECISION: 0.20

APPLICABILITY: The working range is 0.02 fiber/mL (2000-L air sample) to 1.25 fibers/mL (400-L air sample). The method measures the asbestos fiber concentration in air samples taken on mixed cellulose ester filters. The method measures effectively asbestos fibers of the smallest diameter (<0.05 µm) but allows comparison of fiber counts to be made with phase contrast light microscopic data when fiber diameters are rigidly defined.

INTERFERENCES: Non-asbestiform varieties of the amphiboles may interfere in the TEM analysis if the individual particles have aspect ratios greater than 3:1. These interferences can only be eliminated by quantitative zone axis electron diffraction analysis. High concentrations of background dust interfere with fiber identification.

OTHER METHODS: NIOSH Method 7400 is the phase-contrast microscopic method designed for use with this method.

REAGENTS:
1. Acetone.*

*See SPECIAL PRECAUTIONS.

EQUIPMENT:
1. Sampler: field monitor, 25-mm, three-piece cassette with 50-mm electrically-conductive extension cowl and cellulose ester membrane filter, 0.8- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.
   NOTE 2: An electrically conductive extension cowl is recommended to reduce electrostatic effects on fiber sampling and during sample shipment.
2. Personal sampling pump, ≥0.5 L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, transmission electron, with electron diffraction and energy-dispersive X-ray capabilities.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, #10 surgical steel, curved blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, carbon-coated.
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, polyurethane, spongy, 12-mm thick.
11. Low-temperature plasma asher.
12. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
13. Vacuum evaporator.
14. Cork borer, No. 5 (8-mm).
15. Pen, waterproof, marking.
16. Reinforcement, page, gummed.

SPECIAL PRECAUTIONS: Acetone is an extremely flammable liquid (flash point = 0 °F) and precautions must be taken not to ignite it. Heating of >1 mL acetone must be done in a fume hood using a flameless, spark-free heat source.

SAMPLING:
1. Calibrate each personal sampling pump with a representative sampler in line [3].
2. Remove the top cover from the end of the cowl extension (open face) and orient sampler face down. Wrap the joint between the extender and monitor body with shrink tape to prevent air leaks. Where possible, especially at low %RH, attach the sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.

4. Sample at 0.5 L/min or greater [8]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E (fibers/mm²), of 100 to 1300 fibers/mm² [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area (A_c = 385 mm²)] for optimum counting precision. Do not exceed 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/mL), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}.$$

   NOTE: A sampling flow rate of 1 to 4 L/min for 8 hrs is appropriate in non-dusty atmospheres containing ca. 0.1 fiber/mL. Dusty atmospheres require smaller sample volumes; ambient, cleaner atmospheres require larger air samples for optimum counting precision. Flow rates up to 16 L/min are permitted [8].
5. Remove the field monitor at the end of sampling. Replace the plastic top cover and small end caps.
6. Ship the samples upright with conductive cowl attached in a rigid container with sufficient packing material to prevent jostling or damage.
   NOTE: Use only special antistatic polystyrene foam in the shipping container to prevent fiber loss from the sampler filter due to electrostatic forces.

SAMPLE PREPARATION:

7. Remove a circular section from any quadrant of the sample filter using a cork borer.
8. Place the excised section on a clean microscope slide. Affix the filter circle to the slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
   NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
   NOTE: The "hot block" clearing technique of Method 7400 may be used instead of step 9.
10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 x 8-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [17].
11. Place the slide containing the collapsed filters into a low-temperature plasma asher. Etch the filter at 100 °C for ca. 2 min at an O_2 pressure of 130 Pa (1 mm Hg).
    NOTE: Plasma ashers may vary. Determine optimum etching time on blank filters before etching samples.
12. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent.
    NOTE: The wicking substrate should be small enough to fit into the petri dish without touching the lid.
13. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish lid and marking with a waterproof marker on the dish lid. Fill the dish with acetone until the wicking substrate is saturated.
    NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

14. Remove about a quarter section of the carbon-coated filter samples using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

CALIBRATION AND QUALITY CONTROL:

15. Determine the TEM magnification on the fluorescent screen:
    a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
       NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [14].
    b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 °C.
    c. Adjust microscope magnification to 10,000X. Measure the distance (mm) between two widely-separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).
       NOTE: The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.
    d. Calculate the true magnification (M) on the fluorescent screen:

$$M = \frac{X \cdot G}{Y}$$

where: X = total distance (mm) between the designated grating lines;
G = calibration constant of the grating replica (lines/mm);
Y = number of grating replica spaces counted along distance "X."

    e. Once calibration has been completed, note the apparent sizes of 0.25 and 3.0 µm on the fluorescent screen. (These dimensions are essentially the diameter boundary limits for counting asbestos fibers by phase contrast microscopy.) Do not count fibers above 3.0 µm diameter. Count fibers below 0.25 µm diameter but do not include this fraction in calculations when correlating TEM data to PCM counts. Do not count fibers <1 µm long [15].

16. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening diameters. Calculate an average field diameter from the data and use this number to calculate the field area for an average grid opening.
    NOTE: A grid opening is considered as one field.

17. Obtain reference selected area electron diffraction (SAED) patterns from standard asbestos materials prepared for TEM analysis.
    NOTE: This is a visual reference technique. No quantitative SAED analysis is required [14].
    a. Ensure that the specimen holder is oriented at 0 °C tilt for SAED pattern inspection.
    b. Center a fiber, focus, and center the smallest field-limiting aperature on the fiber. Use a 20-cm camera length and 10X binocular head. Obtain an SAED pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.
       NOTE: Not all fibers will present SAED patterns; however, the objective lens current may need to be adjusted to give optimum pattern visibility.

18. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 µm of each asbestos variety obtained from standard reference materials [14].
    - a. Prepare TEM grids of all asbestos varieties.
    - b. Use acquisition times (up to 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥500 counts per channel.
    - c. Estimate the elemental peak heights visually as follows:
        - (1) Normalize all peaks to silicon (assigned an arbitrary value of 10).
        - (2) Visually interpret all other peaks present and assign values relative to the silicon peak.
        - (3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [14].
        - (4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

19. Perform an SAED pattern inspection on all fibers counted under the TEM, using the procedures given in step 16. Inspect the SAED pattern and assign it to one of the following:
    - a. chrysotile;
    - b. amphibole;
    - c. ambiguous;
    - d. none.
20. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 17. Using the SAED pattern and EDX spectrum, classify the fiber:
    - a. For a chrysotile structure, obtain EDX spectra on the first five fibers; then one out of ten thereafter. Label profiles of 0-5-10-0-0- to 0-10-10-0-0 as "chrysotile."
    - b. For an amphibole structure, obtain EDX spectra on the first 10 fibers; then one out of ten thereafter. Label profiles 0-2-10-0-7 as "possible amosite"; profiles 1-1-10-0-6 as "possible crocidolite"; profiles 0-4-10-3-<1 as "possible tremolite"; and profiles 0-3-10-0-1 as "possible anthophyllite."
    - c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."

      NOTE: Fibers smaller than 0.2 µm in diameter may not produce sufficient peak heights to allow an elemental profile to be determined. These fibers should be identified as "probable" asbestos fibers, if SAED patterns are not determinable.
21. Counting and Sizing:
    - a. Insert the sample into the specimen grid holder and scan the grid at 0 °C tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over approximately 75% of the grid openings.
    - b. Estimate the number of fibers per grid opening during the low magnification scan (ca. 1000X). Use the following rules when picking grid openings to count [14]:
        - (1) Light (<5 fibers per opening): count every grid opening in which the carbon film is intact.
        - (2) Moderate (5 to 25 fibers per opening): count every fifth grid opening. If the carbon film is damaged, proceed to the next available opening in which the film is intact.
        - (3) Heavy (>25 fibers per opening): count every tenth grid opening. If the carbon film is damaged, proceed to the next available opening in which the carbon film is intact.

c. Increase magnification to 10,000X. Begin counting at one end of the specimen grid and systematically traverse the grid by rows, reversing direction at row ends.

d. Size all asbestos fibers using a scale inscribed or overlaid on the fluorescent screen.

NOTE: Data can be recorded directly off the screen in mm and later converted to µm by computer. Count and record asbestos fibers and structures >1 µ long of all dimensions [15]. However, when comparing TEM data to the PCM counts, only those fibers greater than 5 µm long and between 0.25 and 3.0 µm diameter which have aspect ratios of 3:1 or greater should be included. These size-adjusted data are necessary to test the comparability of counts between the two methods. If the size-adjusted counts between the two methods show reasonable equivalency, then further analysis using the small-fiber data will show what fraction of airborne asbestos fibers is being missed by the PCM method.

e. Count fibers and asbestos structures using the following rules:

(1) Count all particles less than 3 µm diameter which meet the definition of a fiber (aspect ratio $\geq 3:1$, with parallel sides).

NOTE: Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.

(2) Use the same counting rules as were used for the PCM method for counting single fibers and small bundles (A or B rules). Record separately asbestos fibers between 1 and 5 µm long [14].

(3) Record other asbestos structures according to the morphological definitions:

NOTE: Combinations of these structures are labeled according to the dominant quality (i.e., fiber, bundle, cluster, matrix).

(a) Bundle (B) - a compact arrangement of parallel fibers in which separate fibers or fibrils may only be visible at the ends or edges of the bundle. Asbestos bundles having aspect ratios of 3:1 or greater and less then 3 µm in diameter are counted as fibers.

(b) Cluster (C) - a network of randomly-oriented interlocking fibers arranged so that no fiber is isolated from the group. Dimensions of clusters can only be rdughly estimated and clusters are defined arbitrarily to consist of more than four individual fibers.

(c) Matrix (M) - one or more fibers attached to or imbedded in a non-asbestos particle.

(4) Count fibers which are partially obscured by the grid.

(5) If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a fiber greater than 5 µm only if more than 2.5 µm of fiber is visible. Otherwise, log the fiber as a "short" fiber, measuring only that portion which is visible.

(6) Size each fiber as it is counted and log the diameter and length (in mm). Sizing of fibers is accomplished by moving the fiber to the center of the screen and reading directly the length of the fiber from the scale inscribed on the screen. The scale is most efficient if it consists of a series of scale inscribed on the screen or partial circles every 2 cm distant from the center. For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the concentric circles until its entire length has been measured. When a fiber has been sized, return to the starting point and continue the traverse to the next fiber.

EVALUATION OF METHOD:

The TEM method has been shown to have an overall precision of 20.1% (s_r) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 11.0% (s_r) [12].

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METHOD WRITTEN BY: James W. Carter; Paul A. Baron, Ph.D.; and David G. Taylor, Ph.D.; NIOSH/DPSE.

APPENDIX H

ELECTROSTATIC CHARGE EFFECTS IN ASBESTOS SAMPLING WITH PLASTIC CASSETTES

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Plastic filter cassettes have been used for sampling various workplace aerosols. Electrostatic charges on the particles and cassettes have been postulated as responsible for some of the anomalous effects noted during sampling, especially for asbestos since asbestos measurement requires a uniform deposit of fibers on the filter surface. Both theoretical and experimental measurements have been made to estimate the effect of electrostatic charges found on cassettes used for asbestos sampling. Recent work by Johnston, Vincent and their co-workers have indicated significant charge levels on asbestos fibers in certain workplaces.

Monodisperse potassium biphthalate/methylene blue particles with known charge levels were generated using a technique developed by John and co-workers. These particles were sampled with 37 mm cassettes and 25 mm cassettes with cowls. Several controlled charge configurations were induced on the cassette surface and the resulting deposit of charged particles observed. Similarly, new cassettes with charges on the surface induced by manufacture and normal handling were also investigated.

Two dimensional model calculations were carried out to simulate the above experiments and predict the resultant distribution of particles on the filter. These calculations predicted the distribution of the deposit noted in the experimental measurements. The results of the experiments and the model will be used to investigate previously collected samples and to evaluate improvements in sampling cassettes and collection techniques.

CONCLUSIONS (TO DATE)

I. TYPICAL PLASTIC CASSETTES USED IN THE PAST CAN CARRY A VERY LARGE LOCALIZED CHARGE (ON THE ORDER OF 10^{10} CHARGES/CM^2) ON THEIR SURFACES.

   a. THIS CHARGE CAN EASILY BE GENERATED BY CONTACT WITH OTHER SURFACES AND STAYS ON THE SURFACE FOR A LONG TIME (HOURS TO DAYS).

II. PARTICLES THAT CARRY A SUFFICIENT CHARGE CAN INTERACT WITH THE CHARGED CASSETTE SURFACES AND DEPOSIT ON THE FILTER AND CASSETTE WALLS IN A NON-UNIFORM MANNER.

   a. PARTICLES WITH A KNOWN ELECTRICAL MOBILITY HAVE BEEN GENERATED AND THE DEPOSITION PATTERN HAS BEEN OBSERVED IN CASSETTES WITH TYPICAL CHARGES ON THEIR SURFACES AND IN CASSETTES WITH WELL CHARACTERIZED CHARGES ON THEIR SURFACES.

   b. A TWO DIMENSIONAL MODEL WAS DEVELOPED TO PREDICT THE DEPOSITION PATTERN ASSUMING POINT CHARGES EXISTING ON THE SURFACE OF THE CASSETTE. THIS MODEL QUALITATIVELY PREDICTS THE DEPOSITION PATTERNS OBSERVED. ACTUAL CHARGES ON CASSETTE SURFACES HAVE A COMPLEX DISTRIBUTION BUT TEND TO BE FAIRLY LOCALIZED.

   c. THE MODEL CAN BE USED TO PREDICT THE SHAPES OF THE DISTRIBUTIONS OF PARTICLES ON FILTERS ASSUMING KNOWLEDGE OF PARTICLE MOBILITY AND CASSETTE SURFACE CHARGE. CONVERSELY, MEASURED PARTICLE DISTRIBUTIONS ON FILTERS CAN BE USED TO ESTIMATE THE COMBINED LEVEL OF PARTICLE AND SURFACE CHARGE.

III. CHARGE EFFECTS IN SAMPLING CAN BE REDUCED BY USING CONDUCTIVE CASSETTES OR BY NEUTRALIZING THE AEROSOL BEING SAMPLED.

   a. CONDUCTIVE CASSETTE MATERIAL WILL DISTRIBUTE TRIBOELECTRICALLY GENERATED CHARGES OVER THE ENTIRE SURFACE, REDUCING THE EFFECT ON PARTICLE TRAJECTORIES.

      1. IF THE CHARGE ON A CONDUCTIVE CASSETTE IS LARGE ENOUGH THE PARTICLE DEPOSIT MAY ALSO BE DISTORTED AND THE PARTICLES WILL BE UNDERSAMPLED ON THE FILTER.

   b. ANTI-STATIC SPRAYS CAN DISTRIBUTE SURFACE CHARGE.

      1. SOME USE A CONDUCTIVE LIQUID THAT EVAPORATES AND LEAVES THE SURFACE VULNERABLE TO SURFACE CHARGE REGENERATION BY TRIBOELECTRIC CONTACT.

      2. OTHER SPRAYS DEPOSIT A SALT THAT RETAINS WATER DEPENDING ON RELATIVE HUMIDITY AND FORMS A SLIGHTLY CONDUCTIVE LAYER. THIS MATERIAL CAN BECOME AS GOOD AN INSULATOR AS THE PLASTIC SURFACE UNDER VERY LOW HUMIDITIES (LESS THAN 20% RH, THOUGH THIS HAS NOT BEEN COMPLETELY INVESTIGATED YET)

   c. RADIOACTIVE SOURCES (SUCH AS PO-210) CAN REDUCE THE CHARGE ON AEROSOL PARTICLES AS WELL AS ON CHARGED CASSETTE SURFACES AND THUS ELIMINATE BIAS AND VARIABILITY IN CHARGED PARTICLE SAMPLING.

APPENDIX I

EXTRACTION PROCEDURE AND INTERLAB COMPARISON OF BLANK ANALYSIS OF AIRBORNE ASBESTOS

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MODIFICATION OF METHOD

The procedure described is a modification of the original method developed for the EPA for measuring airborne asbestos fibers (Samudra et al. 1978). The word "Airborne" constitutes the free movement of any particle or fiber material, i.e., matter from ambient air. Overall the concentration of particles, collected within a given volume of air and deposited onto a filter substrate from a workplace environment, has been applied to the sampling methodology section of the provisional protocol of the EPA.

The purpose of the preparation step is to transfer particles from a filter surface to an EM grid with a minimum amount of distortion in morphology and size distribution. To date only a few improvements have been made using the current technique due to preparation problems in standardizing the present method. For that reason a modified version is recommended for consideration. The technique presented here was conceived in the U.S. Environmental Protection Agency's EM Facility and has proven to be very valuable, time and cost effective, and less tedious, omitting several steps during the procedure.

PREPARATION

The basis for the method is to transfer the collected particulates from the carbon-coated polycarbonate filter to an EM 200 MESH carbon-coated grid. Before the transfer is made the preparation of all materials is necessary to insure a smooth operation during the technique. Figure 1 shows the layout of the 9cm Whatman filter paper currently in use. Next a 2 inch square cross section is drawn directly onto the filter paper with a No. 2 lead pencil allowing up to 25 1/2" grid block preparations. Two 0.5 inch V-shaped notches are cut into the filter paper parallel to the sides of the cross section, creating adjacent wells for adding solvent (chloroform). Labelling of each cell area is done directly on the filter paper at this time. Figure 2 shows the V-shaped multiple-box arrangement. The technique for cutting filters involves a 9mm carbon-coated polycarbonate section excised from the center of each cassette. Using the pointed tip of a sharp scapel. A rectangular section may be cut by making 4 direct paths that intersect across the exposed filter area. The result is shown in Figure 3. All remaining cassettes should be cut and stored in this manner Figure 3a. The Jaffe Wick washer (glass petri dish) is made airtight by using a carborondum powder (80 MESH) with water to ground a flat surface on the rim of the bottom dish and an opposing recessed surface on the underside of the upper dish. This creates a ground-glass seal. The use of this step provides a significant reduction in the evaporation process of the solvent over a longer period of time.

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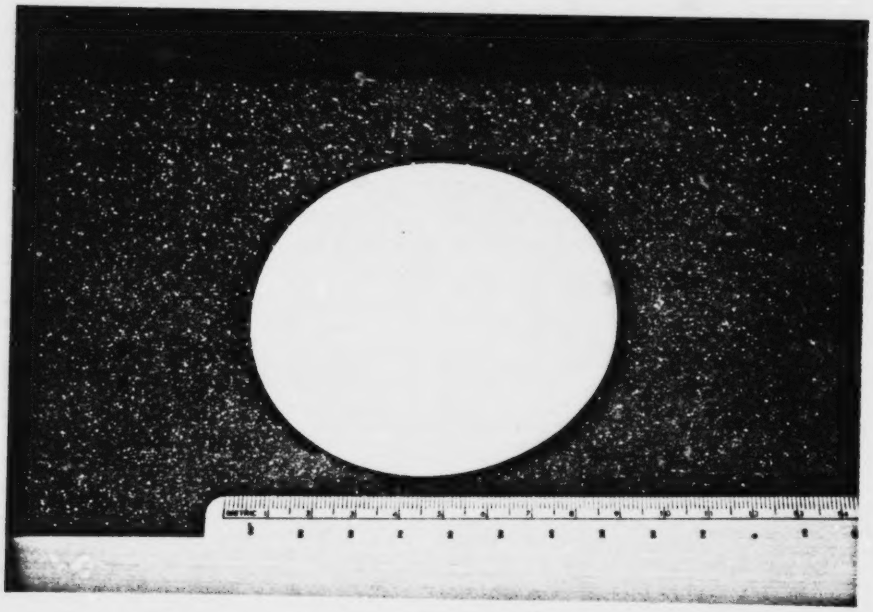

FIGURE 1

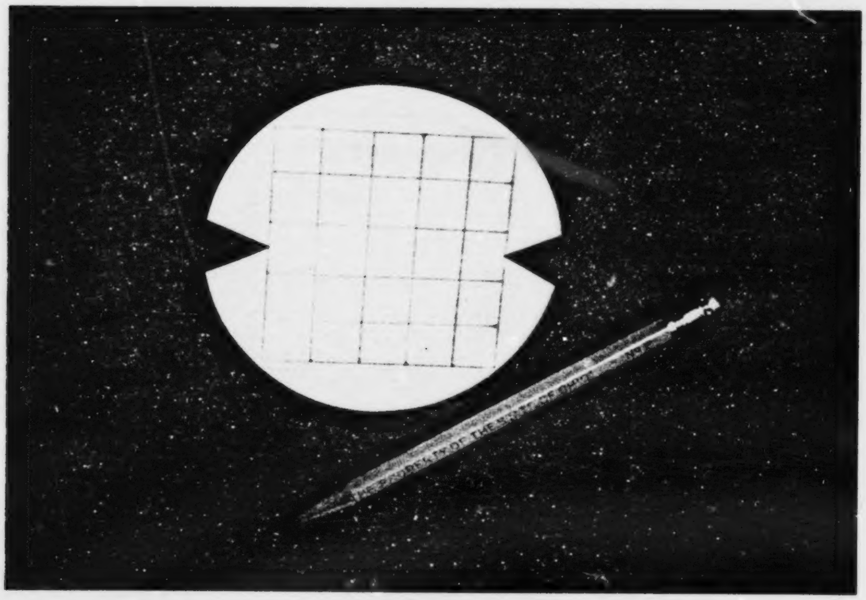

FIGURE 2

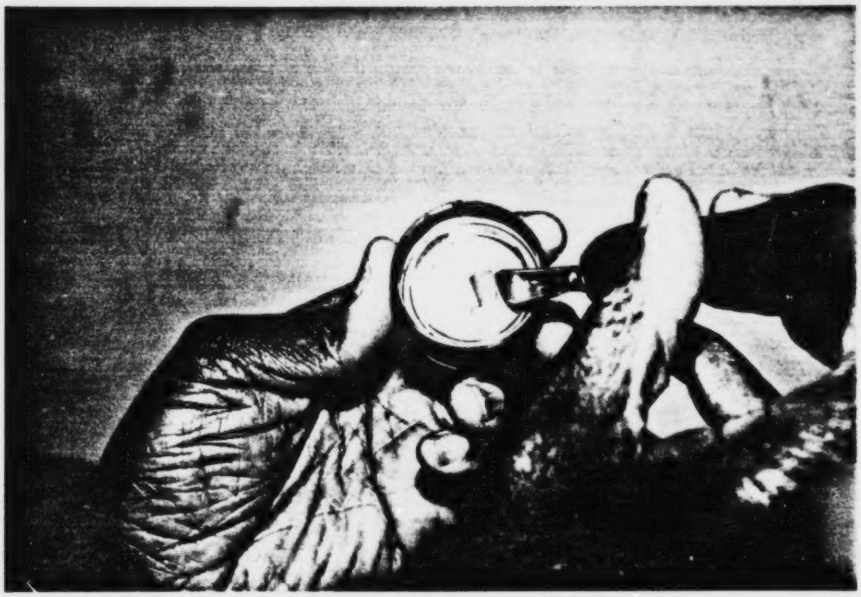

FIGURE 3

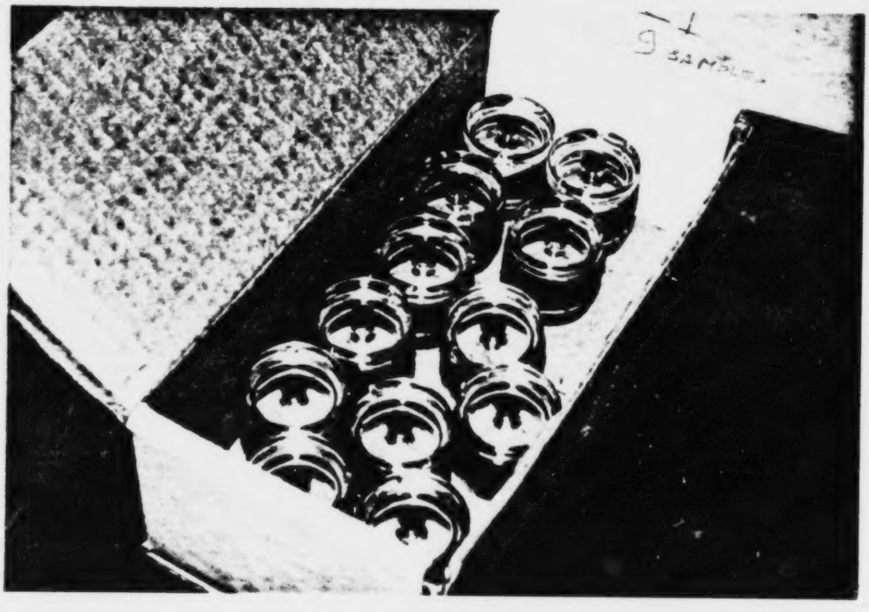

FIGURE 3a

PROCEDURE

A combination of polyurethane foam and a single sheet of Whatman filter paper (refer to Figure 2) both 9cm in diameter are used as the substrate support. Fill the lower half of the petri dish with spectrographic-grade chloroform (solvent) throught the V-shaped port areas until the foam swells causing the level of the liquid to impact on the surface of the filter. On top of the filter, pieces of 100-MESH stainless steel screen (1.0cm x 1.0cm) are positioned in each individual cell block with one 200-MESH carbon-coated EM grid placed at the center location of each screen. A filter section is then removed (Figure 4) with the tweezers and carefully laid, carbon side down, on the damp 100-MESH stainless steel grid screen substrate. Figures 5 and 6 illustrate this method excluding the choloroform bath. The EM rid-filter-screen combination will immediately "wet out" and remain on the screen. Note, the electron-charged particles deposited on the polycarbonate filter will sometimes cause it to curl up from conductivity and become distorted. To allow for better contact try to avoid filter segments that are not completely flat. Once all specimens are placed in the Jaffe Wick washer (Figure 7) more chloroform is carefully added as needed to maintain the appropriate level of solvent. The filter becomes completely dissolved (cleared) by the wicking action in 48 to 72 hours. An additional weight (Figure 8, 500ml of chloroform) resting on top of the petri cover will minimize the chloroform vapor loss. If solvent condensation should occur on the undersurface, a small wedge can be placed at the base of the Jaffe washer allowing the condensation drops to flow out toward the lower edge. Room temperatures lower than 20 degrees celcius (68 degrees F) may take longer than 72 hours to completely dissolve the filters. When the filters have reached

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FIGURE 4

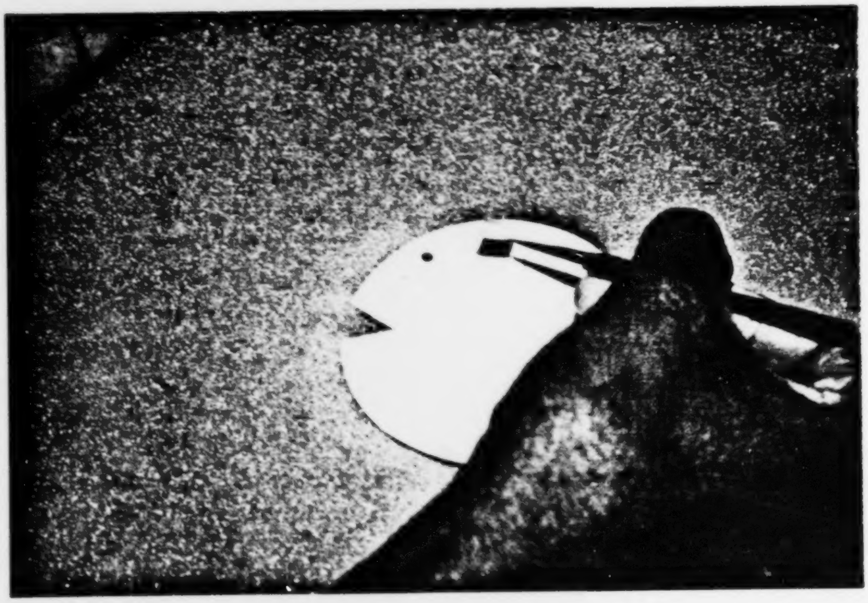

FIGURE 5

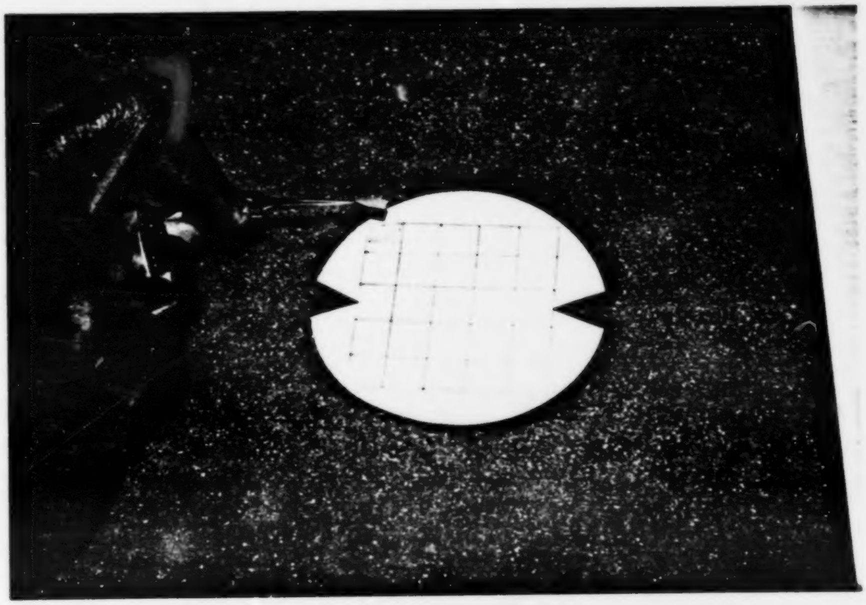

FIGURE 6

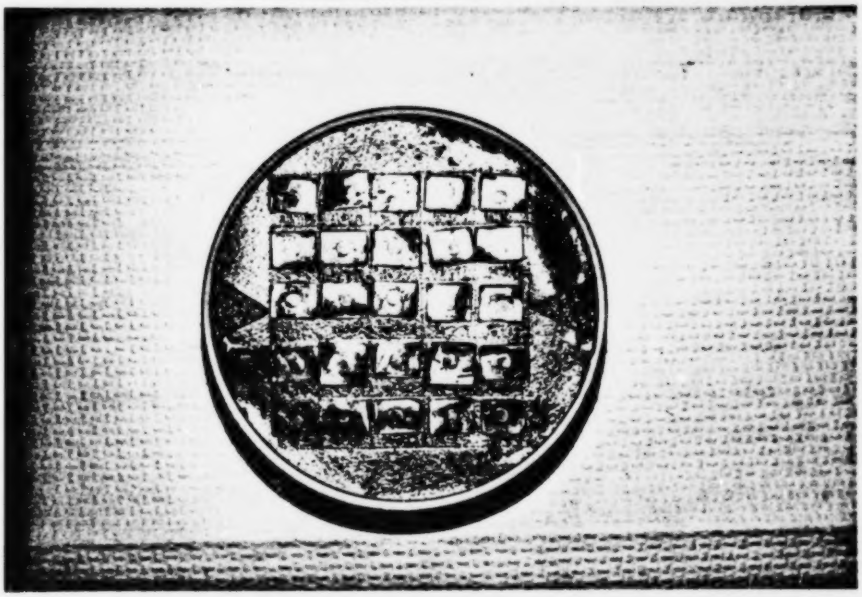

FIGURE 7

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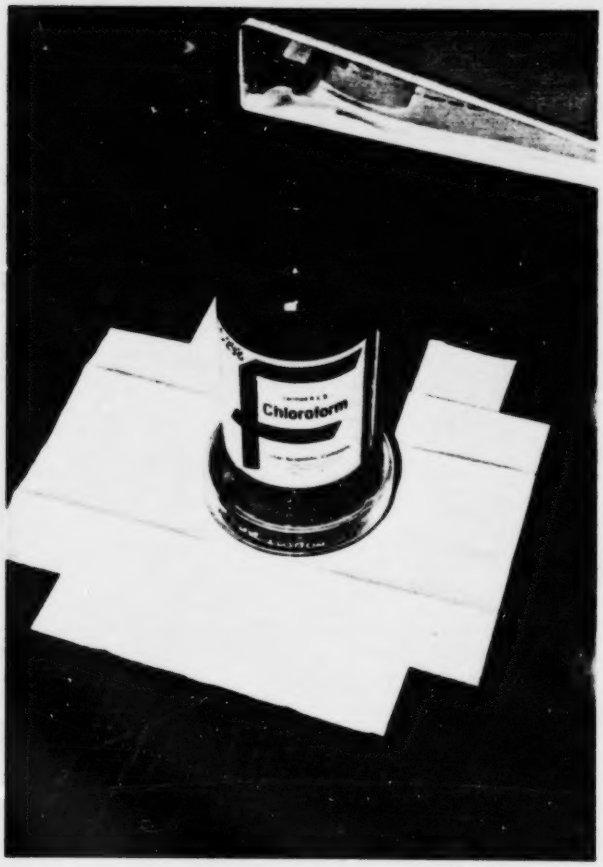
Chloroform

FIGURE 8

complete clearing, the grid is removed and stored in the gridbox and identified by code number in the logbook.

CONCLUSIONS

Figure 9 illustrates the Jaffe Wick Washer, gridbox, capsule and grid, No. 2 lead pencil, cell block diagram, spectrographic-grade chloroform, V-shaped notches, scapel, nuclepore polycarbonate filter, polyurethane foam, filter paper, and stainless steel tweezers. Several considerations should be observed to improve the modifications of the current transmission electron microscopy technique. This method appears to be better, and more efficient than the procedure previously recommended.[1]

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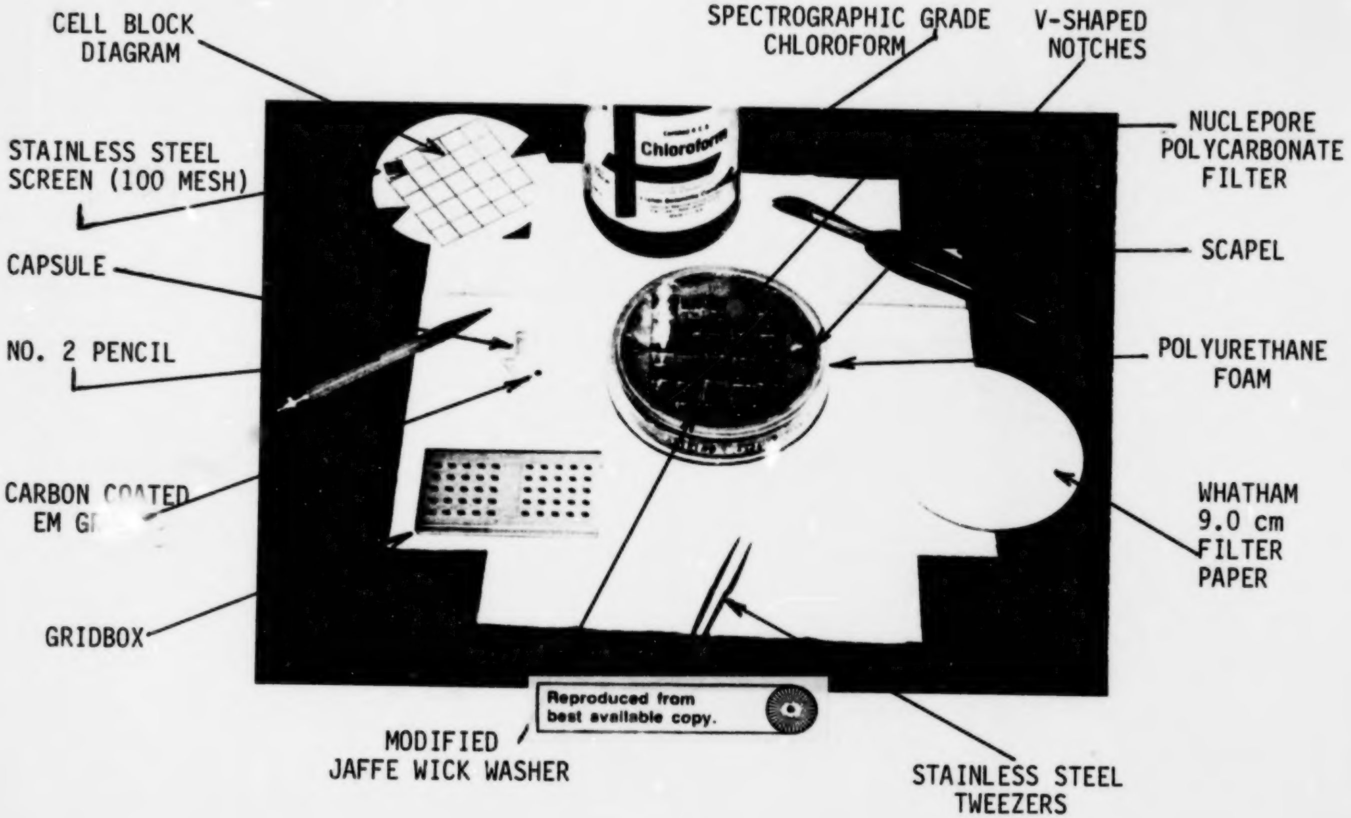
CELL BLOCK DIAGRAM
SPECTROGRAPHIC GRADE CHLOROFORM
V-SHAPED NOTCHES
STAINLESS STEEL SCREEN (100 MESH)
NUCLEPORE POLYCARBONATE FILTER
CAPSULE
SCAPEL
NO. 2 PENCIL
POLYURETHANE FOAM
CARBON COATED EM GR
WHATHAM 9.0 cm FILTER PAPER
GRIDBOX
Chloroform
MODIFIED JAFFE WICK WASHER
STAINLESS STEEL TWEEZERS

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FIGURE 9

INTERLAB COMPARISON OF BLANK ANALYSIS

COMPARISON OF TEM ANALYSES

Using the data from the asbestos blank study, a comparison was made on the number of fibers found between the labs. The data consisted of six parts and four labs (see data sheet). The analysis was performed for all three categories of fibers - asbestos, nonasbestos, and ambiguous - and for the total number of fibers found.

Analysis of variance statistical procedure was used with the main effects of lab and part. The initial analysis indicated that there was no significant differences among the six parts, so the analysis was rerun with just the lab effect.

The results of the analysis indicated that there was a significant difference among the labs with respect to each category of fibers (asbestos, p-value= .0001; nonasbestos, p-value= .0001; ambiguous, p-value= .0013; and total, p-value= .0001). A comparison of means showed that McCrone found a significantly higher amount of asbestos fibers than any other lab. The EPA found the greatest number of nonasbestos fibers, while NIOSH found significantly more nonasbestos fibers than either ETC or McCrone. The EPA and NIOSH found ambiguous fibers while ETC and McCrone found none. Overall, the EPA found a significantly higher amount of fibers than any other lab, while ETC found a significantly lower amount of fibers than any other lab.

DISCUSSION

Overall EPA's analyses for asbestos were slightly lower than ETC's analyses. There appear to be two reasons for this. First, the EPA analyst has had little experience with amphiboles, so he was not familiar with their electron diffraction patterns. Second, EPA had no energy dispersive system on their JOEL 100B where the analyses were made. These mitigating circumstances made it impossible to positively identify some amphiboles and all unknown crystal patterns were reported in the ambiguous column. Many of these may have been amphibole fibers.

McCrone appears to have had a contamination problem as their analyses were quite high compared to the other laboratories. Also, the analysts at NIOSH have not had as much fiber identification experience as the other analysts and may have missed the extremely small fibers found, the majority of which were under .5 microns in length. The NIOSH grids had a higher percentage of undissolved filter which may have been due to not using the screens in the Jaffe Wick procedure (the grids were placed right on the filter). After using the screens, the amount of extaneous filter decreased dramatically. This would enable the smaller fibers to be seen more easily.

The nonasbestos fiber observations are the easiest to explain. The fibers were definately undissolved filter that appeared fibrous but had slightly unparallel ends. The data suggest that

NIOSH's and EPA's counts were close. Again the high amount of nonasbestos fibers appears to be due to not using a screen in the dissolving portion of the procedure. Using a screen does improve the wicking action. Outside of these nonasbestos fibers there were almost no nonasbestos fibers which would put EPA and NIOSH very close to ETC and McCrone.

In conclusion, there appears to be some contamination in the nucleopore method. The fibers seem to be of consistant number and also of consistant size (the average length less than .5 micrometers). In a study with large numbers of fibers and numerous sizes, it would have a negative effect. If there are only a few fibers on the filter sample, the effect would be enormous.

TABLE 1
AVERAGE NUMBER OF FIBERS

ASBESTOS		NONASBESTOS		AMBIGUOUS		TOTAL	
McCrone	21.2	EPA	23.8	EPA	2.3	EPA	30.0
ETC	7.5	NIOSH	13.8	NIOSH	1.5	McCrone	21.2
EPA	4.0	ETC	1.2	ETC	0.0	NIOSH	15.7
NIOSH	0.3	McCrone	0.0	McCrone	0.0	ETC	8.7

TABLE 2
ASBESTOS

Part/Lab	McCrone	NIOSH	ETC	EPA1
W-1	22	0	6	5
W-2	33	0	3	3
W-3	17	2	15	-
P-1	20	0	15	5* (7)
P-2	16	0	0	1
P-3	19	0	6	-

TABLE 3
NONASBESTOS

Part/Lab	McCrone	NIOSH	ETC	EPA1
W-1	0	20	1	18
W-2	0	9	1	22
W-3	0	8	5	-
P-1	0	16	0	16* (23)
P-2	0	16	0	32
P-3	0	14	0	-

TABLE 4

AMBIGUOUS

Part/Lab	McCrone	NIOSH	ETC	EPA1
W-1	0	1	0	2
W-2	0	3	0	4
W-3	0	1	0	-
P-1	0	0	0	2* (3)
P-2	0	2	0	0
P-3	0	2	0	-

APPENDIX J

CURRENT PROCEDURES FOR HANDLING ASBESTOS DATA FROM TEM ANALYSIS

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John Menkedick
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Section 1

Asbestos data collected by air sampling through nucleopore filters and analyzed by TEM has been received at the Cincinnati EPA either on handwritten laboratory data record sheets or on an IBM PC compatible diskette. If received on handwritten laboratory sheets, the data is entered by a data entry clerk directly on a public work disc for the IBM 3081 mainframe at the National Computer Center. If received on an IBM PC compatible diskette the data file is checked and edited to insure that it is in the format outlined in Section 2, then uploaded from the PC to the IBM mainframe at NCC. In either case certain specified information must accompany each sample's data. Since data entry hours are not available for the routine entry of asbestos data from lab sheets, it is almost always required that the data be submitted on diskettes. Requirements for the necessary information and specified format for the data when submitted on a diskette are given in detail in Section 2.

Exhibit 1 presents the output from a single sample stored on a diskette.

Exhibit 2 presents a sample laboratory's data sheet containing the necessary information for handwritten submissions.

Once the data has been made available to the IBM mainframe it is read into a SAS program and converted to a SAS dataset and work file. At this point, it is standard practice to plot the data for each individual sample using the SAS GRAPH graphics procedure. A sample plot is given in Exhibit 3. Plots of the data for any different combination of samples (all the ambient samples, all nonaggressive samples, all blanks, etc) can also be generated. Using SAS, summary statistics can be calculated, the data tabulated and analyzed statistically for outliers or differences of interest (e.g. differences in sampling method — aggressive or nonaggressive).

Section 2

Required Information and Formats for Reporting Asbestos Data from TEM Analysis

The data must be entered on an IBM PC compatible floppy diskette in a Lotus worksheet and stored in a lotus print file. If lotus is not available the data may be submitted in an ASCII sequential file identical to a lotus print file.

For each sample the following information must be given in a format identical to that presented in Lotus (see Exhibit 1B).

For each sample the following information must be given:

1. The lab's name.
2. A unique sample number.
3. The date the sample was taken.
4. The location of the sample.
5. The time of the sample (preabatement or postabatement).
6. The type of sampling (aggressive or passive).
7. Where the sample was taken (inside, outdoors, field blank or quality control).
8. The volume sampled.
9. The date the sample was analyzed.
10. The analyst's name or initials.
11. The magnification.
12. The volume analyzed.
13. The grid square area.
14. The filter type.
15. The filter area.
16. The number of grids examined.

For each particle analyzed within a sample, the following information must be given:

17. The particle number.
18. The grid square number.
19. The type of particle (chrysotile, amphibole, nonasbestos, or ambiguous).
20. The structure of the particle (fiber, bundle, cluster, matrix).
21. The screen length of the particle (dimensions should be included).
22. The screen width of the particle (dimensions should be included).

EXHIBIT 1

LAB	SAMPLE #	DATE SAMP	LOCATION	TIME SAMP (pre,post)	TYPE SAMP (agg,pass)
McCrone	ECEN 1819	6/21/86	Winton ps	pre	agg

WHERE SAMP (i,o,f,q)	VOL SAMP	DATE ANAL	ANALYST	MAG	VOL ANAL
i	1425	6/25/86	jym	20000	1425

GD SQ AREA	FIL TYPE	FIL AREA	# GD EXAM
8100	nucleopore	855	10

PARTICLE #	GRD SQ	TYPE (c,a,n,q)	STRUCTURE (f,b,c,m)	LENGTH (cm)	WIDTH (cm)
1	1	a	f	2.2	0.3
2	3	c	b	0.6	0.2
3	3	c	b	1.5	0.3
4	6	c	b	1.5	0.3
5	6	c	b	1.7	0.3
6	7	a	f	5.7	0.2
7	7	c	b	1.5	0.4
8	7	a	f	0.9	0.2
9	8	c	c	1.9	0.3
10	8	c	b	2.2	0.3
11	8	a	f	1.3	0.2
12	8	a	f	1.0	0.2
13	9	c	b	1.3	0.2
14	9	c	f	0.8	0.2
15	10	c	b	1.9	0.2
16	10	c	b	1.4	0.3
17	10	a	f	2.3	0.3

EXHIBIT 1B

LAB	SAMPLE #	DATE SAMP	LOCATION	TIME SAMP (pre,post)	TYPE SAMP (agg,pass)
1	2	3	4	5	6
WHERE SAMP (i,o,f,q)	VOL SAMP	DATE ANAL	ANALYST	MAG	VOL ANAL
7	8	9	10	11	12
GD SQ AREA	FIL TYPE	FIL AREA	# GD EXAM		
13	14	15	16		
PARTICLE #	GRD SQ	TYPE (c,a,n,q)	STRUCTURE (f,b,c,m)	LENGTH	WIDTH
17	18	19	20	21	22

EXHIBIT 2

LAB ______________
SAMPLE # _______________
DATE SAMPLED ____________
LOCATION _______________
TIME SAMPLED (pre,post) _________
TYPE SAMPLE (agg,pass) ___________
PLACE SAMPLED (in,out,fb,qc) ______
VOLUME SAMPLED _____________

DATE ANALYZED ______________
ANALYST ______________
MAGNIFICATION ____________
VOLUME ANALYZED ____________
GRID SQUARE AREA _____________
FILTER TYPE __________________
FILTER AREA _____________
GRIDS EXAMINED _________

PARTICLE #	GRID SQUARE	TYPE (c,a,n,q)	STRUCTURE (f,b,c,m)	LENGTH (m)	WIDTH (m)

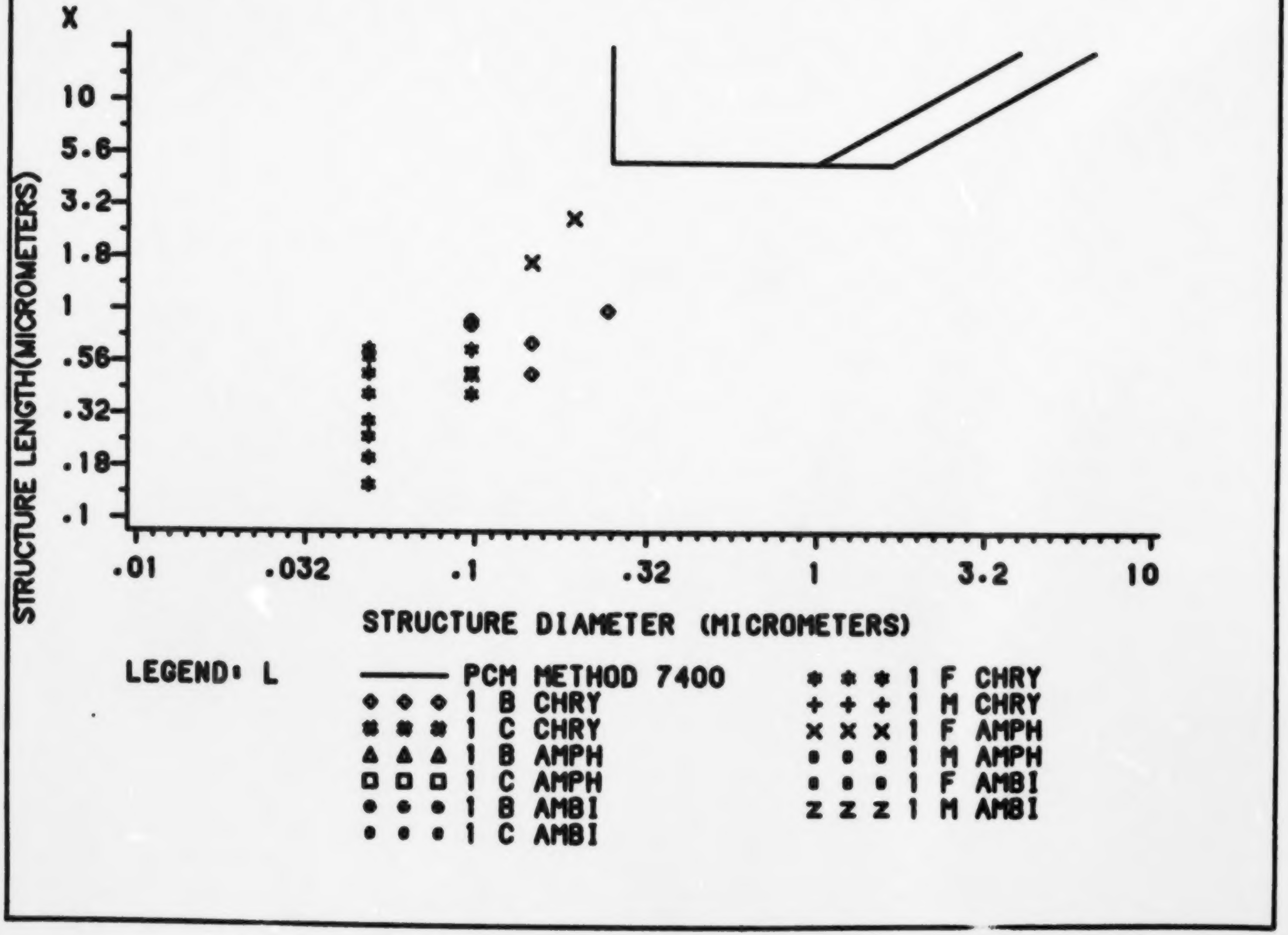
```
McCrone# PEI #    Date Samp  # Part.   Mag   Analyst
 1819174 ECEN1255 2/15/86        13     9200 JJS

Grd sq A Filt A   Vol.samp Vol.anal No. G sq Str./M3
    7200      855                        10

Feature  Grd sq # Type      Structure Screen Measurement Actual Fiber Size
                  c,a,n,q   f,b,c,m   len. cm width cm   len. um  width um

      1         1 c         f               1.0      0.10       1.09     0.109
      2         3 c         b               0.7      0.10       0.76     0.109
      3         3 c         b               1.2      0.10       1.30     0.109
      4         3 c         b               0.9      0.10       0.98     0.109
      5         3 c         f               0.2      0.05       0.22     0.054
      6         5 c         f               0.4      0.05       0.43     0.054
      7         5 a         ?               0.7      0.20       0.76     0.217
      8         5 c         f               0.3      0.05       0.33     0.054
      9         6 c         b               0.9      0.20       0.98     0.217
     10         6 c         f               0.6      0.10       0.65     0.109
     11         8 a         f               0.4      0.10       0.43     0.109
     12        10 c         f               0.6      0.10       0.65     0.109
     13        10 c         f               0.7      0.10       0.76     0.109
```

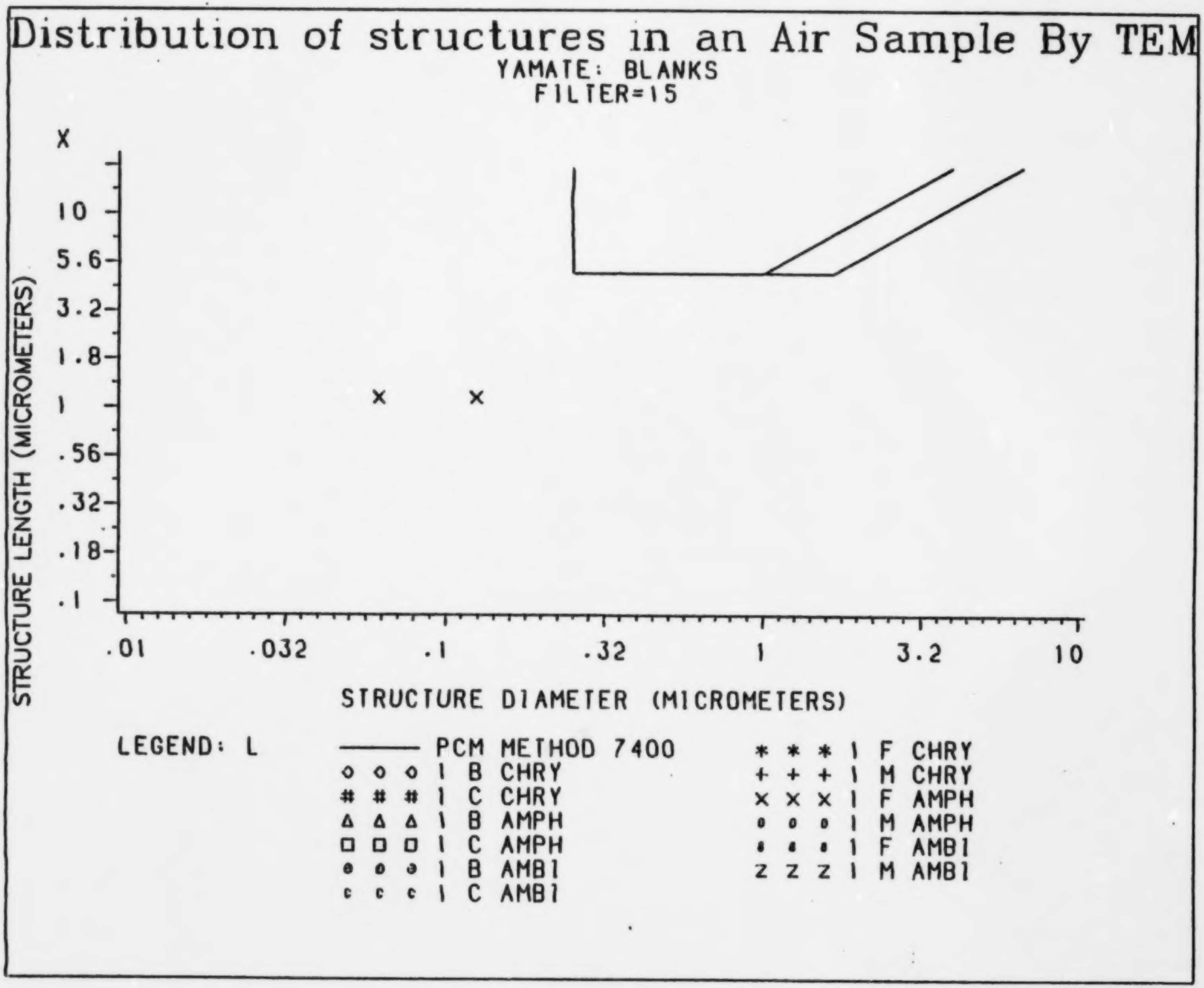
EXHIBIT 3



Distribution of Structures in an Air Sample by TEM

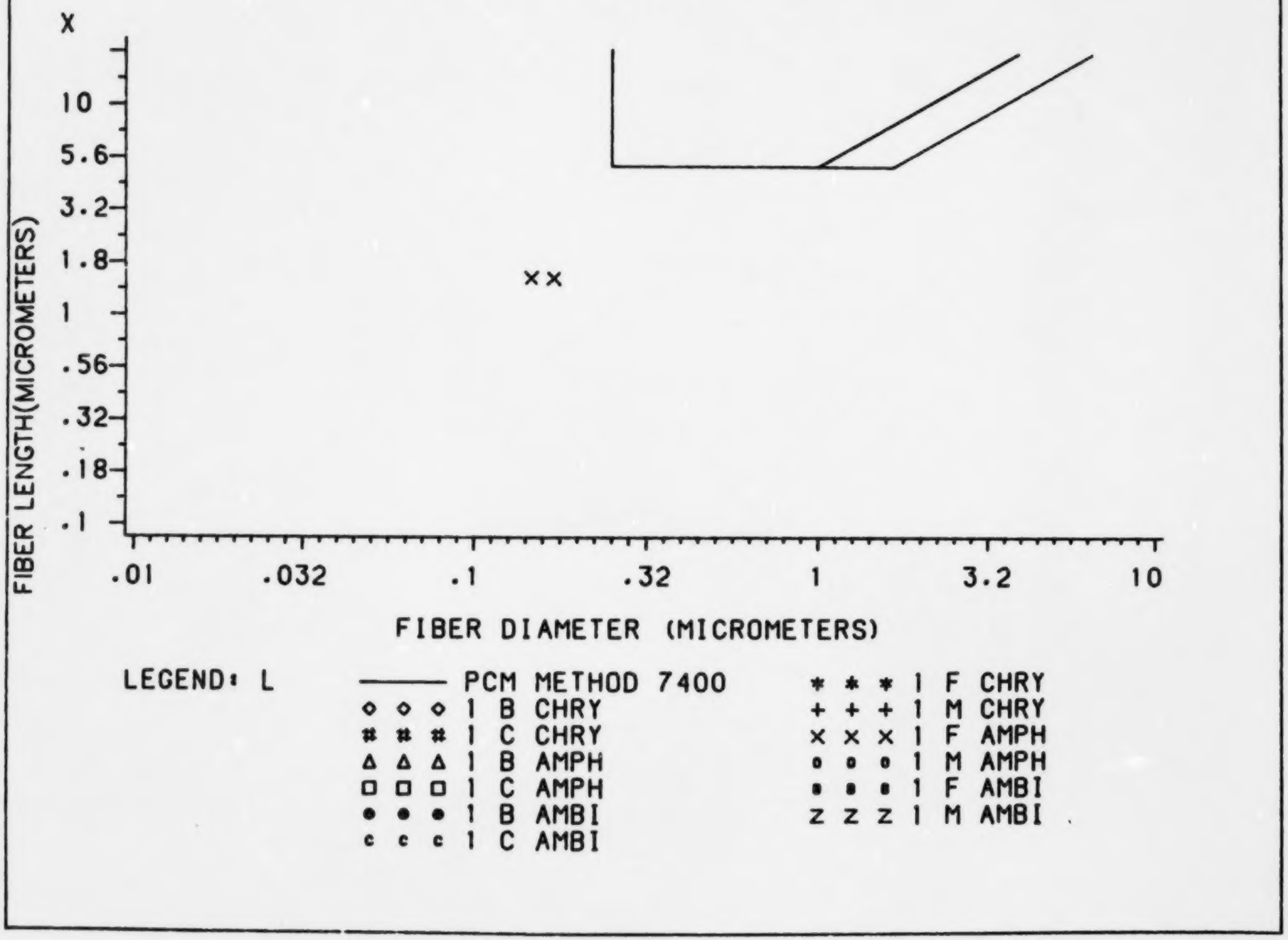
MCCRONE: W1B-W3B & P1B-P3B

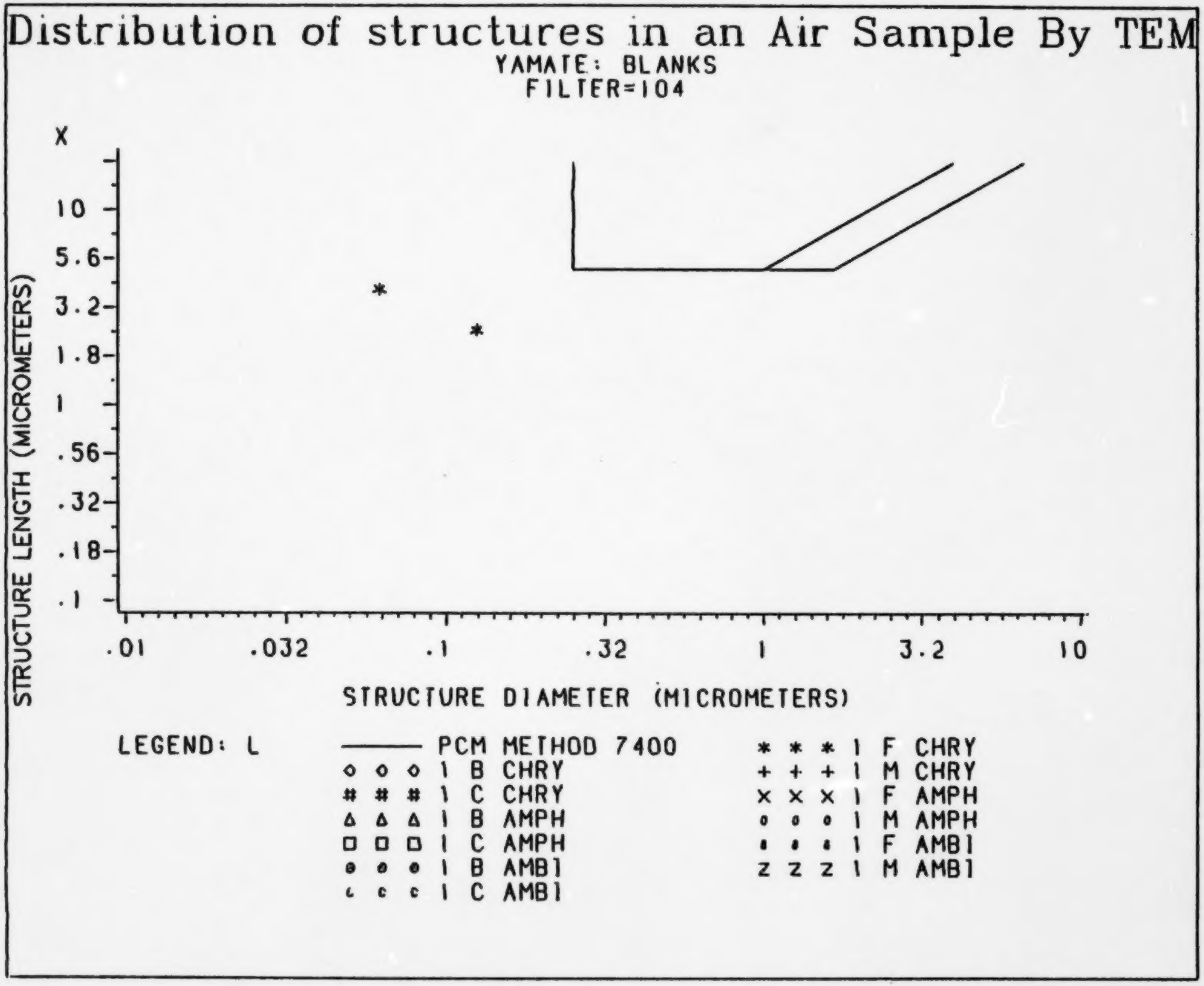
FILTER=W1B


Distribution of structures in an Air Sample By TEM
YAMATE: BLANKS
FILTER=15
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
STRUCTURE LENGTH (MICROMETERS)
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
——— PCM METHOD 7400
◇ ◇ ◇ 1 B CHRY
# 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
ø ø ø 1 B AMBI
c c c 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
○ ○ ○ 1 M AMPH
• • • 1 F AMBI
z z z 1 M AMBI

Size Distribution of Fibers in an Air Sample By TEM

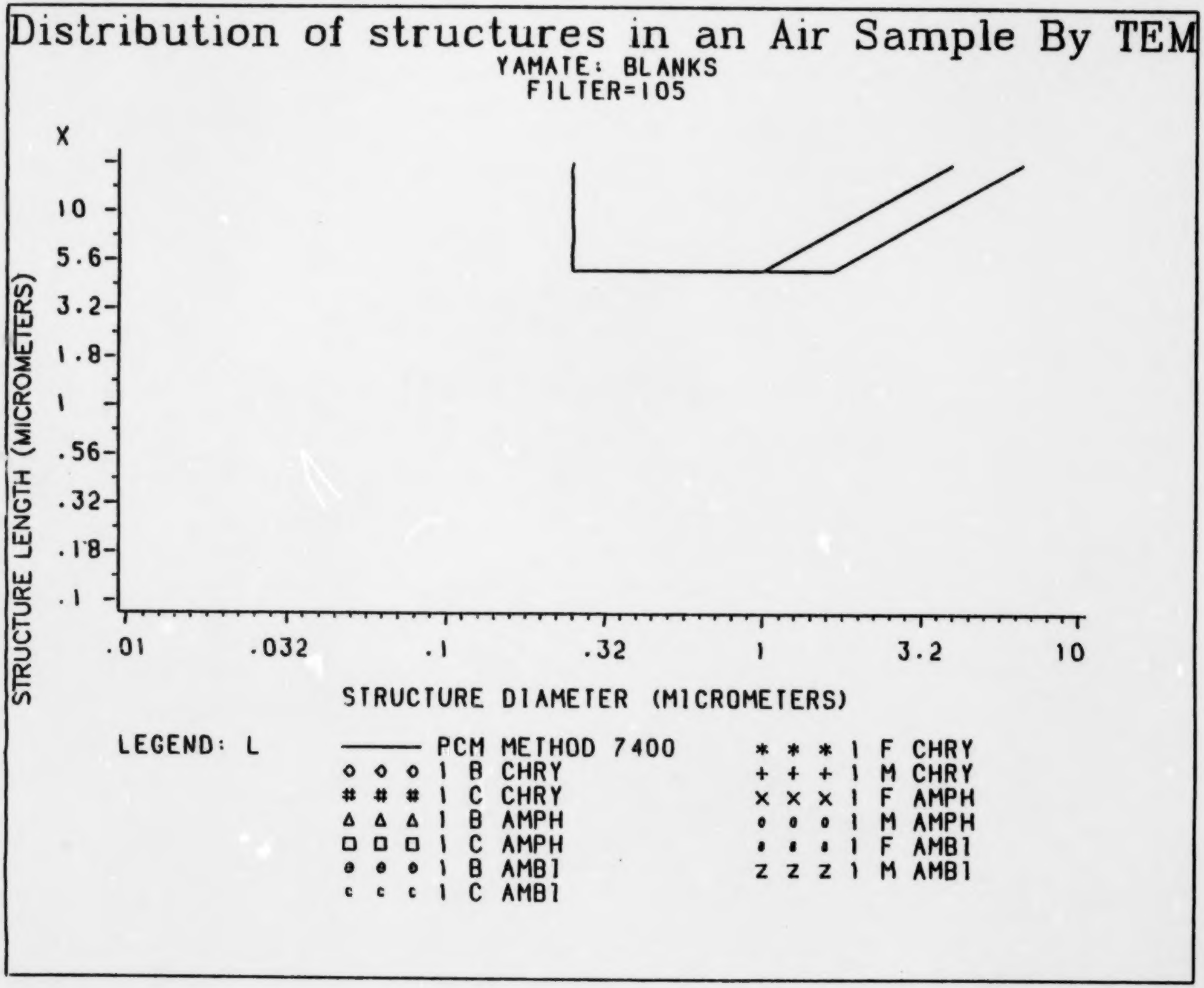
MCCRONE: COLUMBUS EAST PHASE 1 BLANKS
FILTER=104





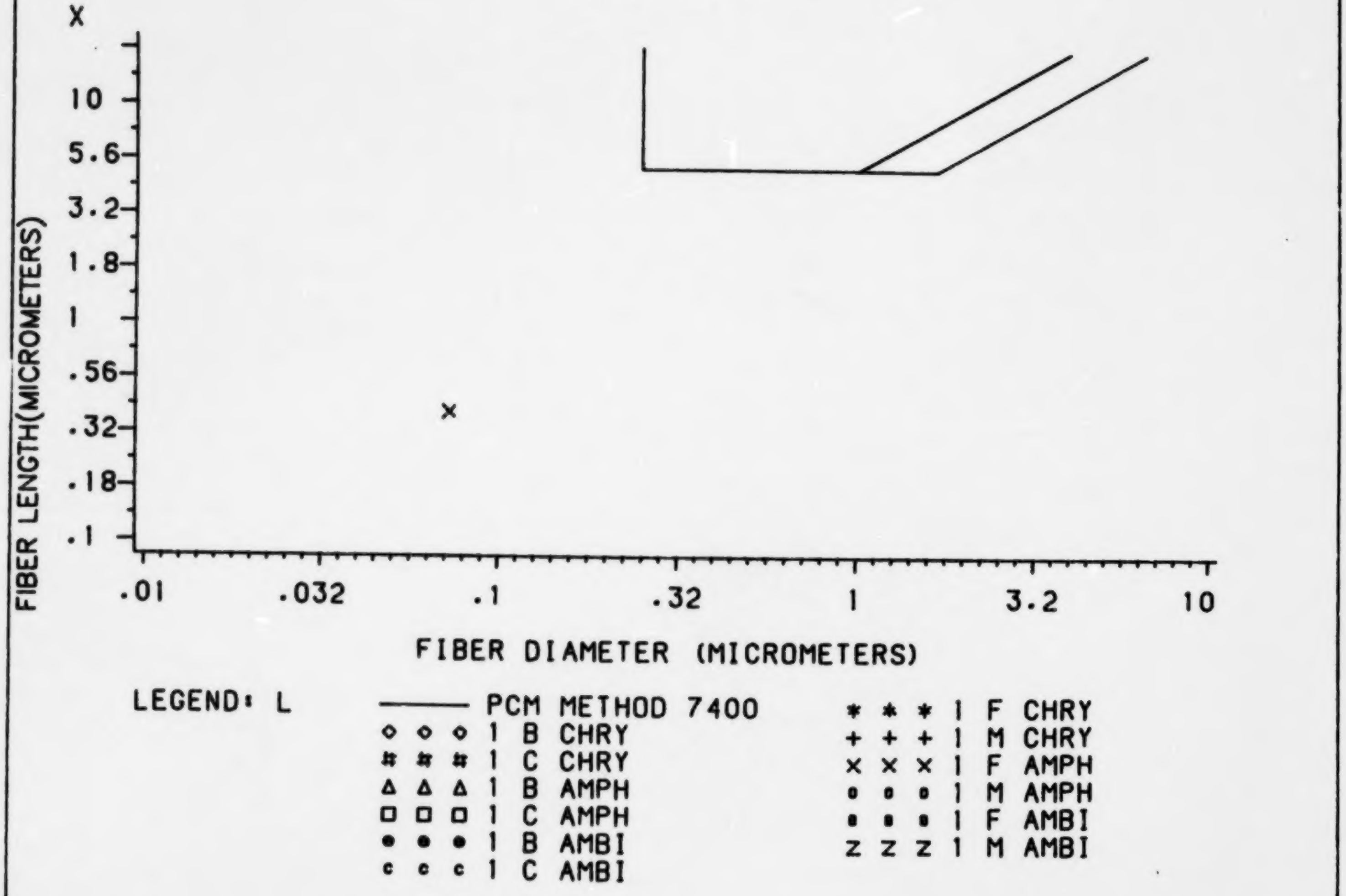
Distribution of structures in an Air Sample By TEM

YAMATE: BLANKS
FILTER=104



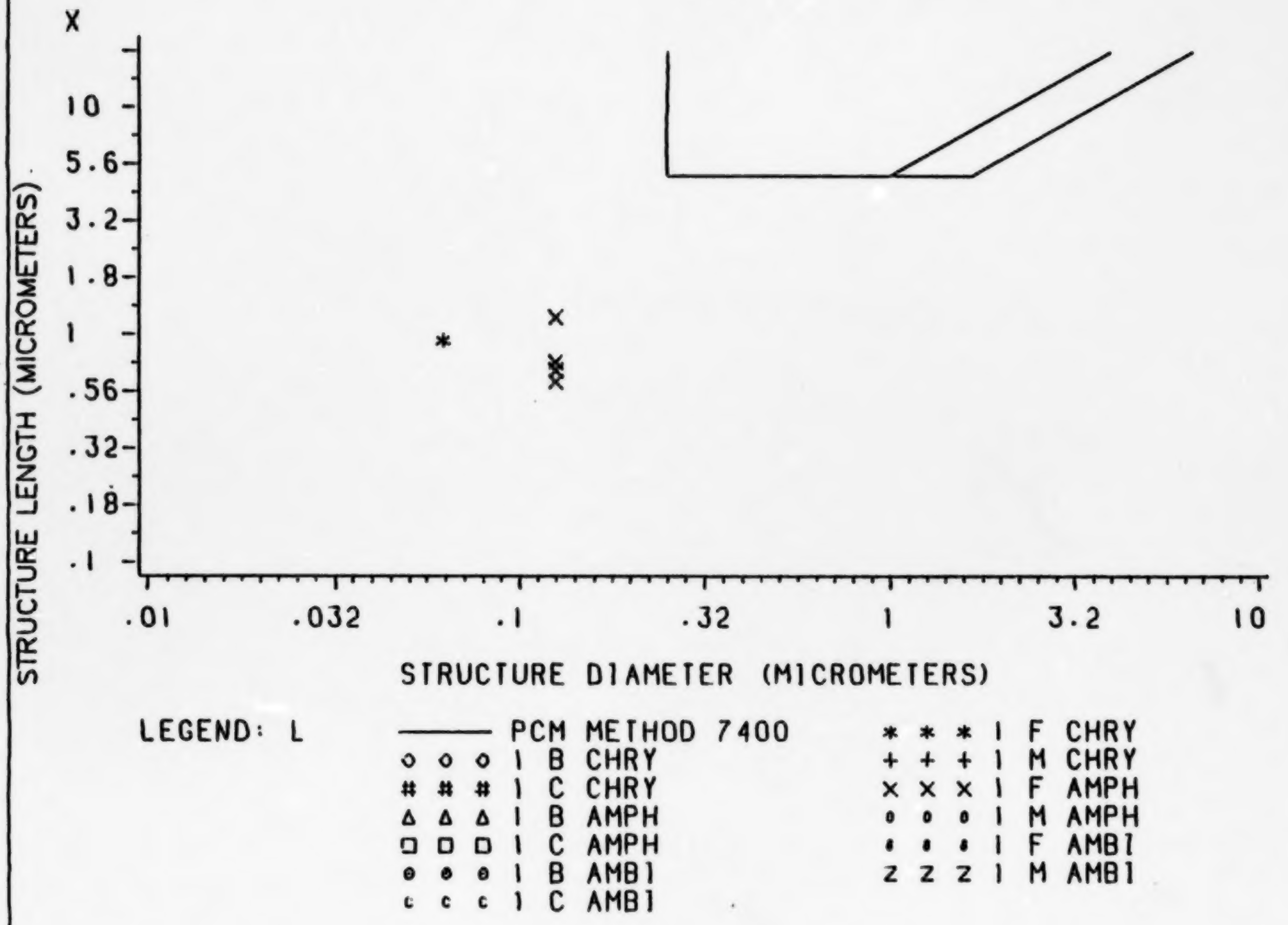
Distribution of structures in an Air Sample By TEM

Size Distribution of Fibers in an Air Sample By TEM
MCCRONE: COLUMBUS EAST PHASE 1 BLANKS
FILTER=106



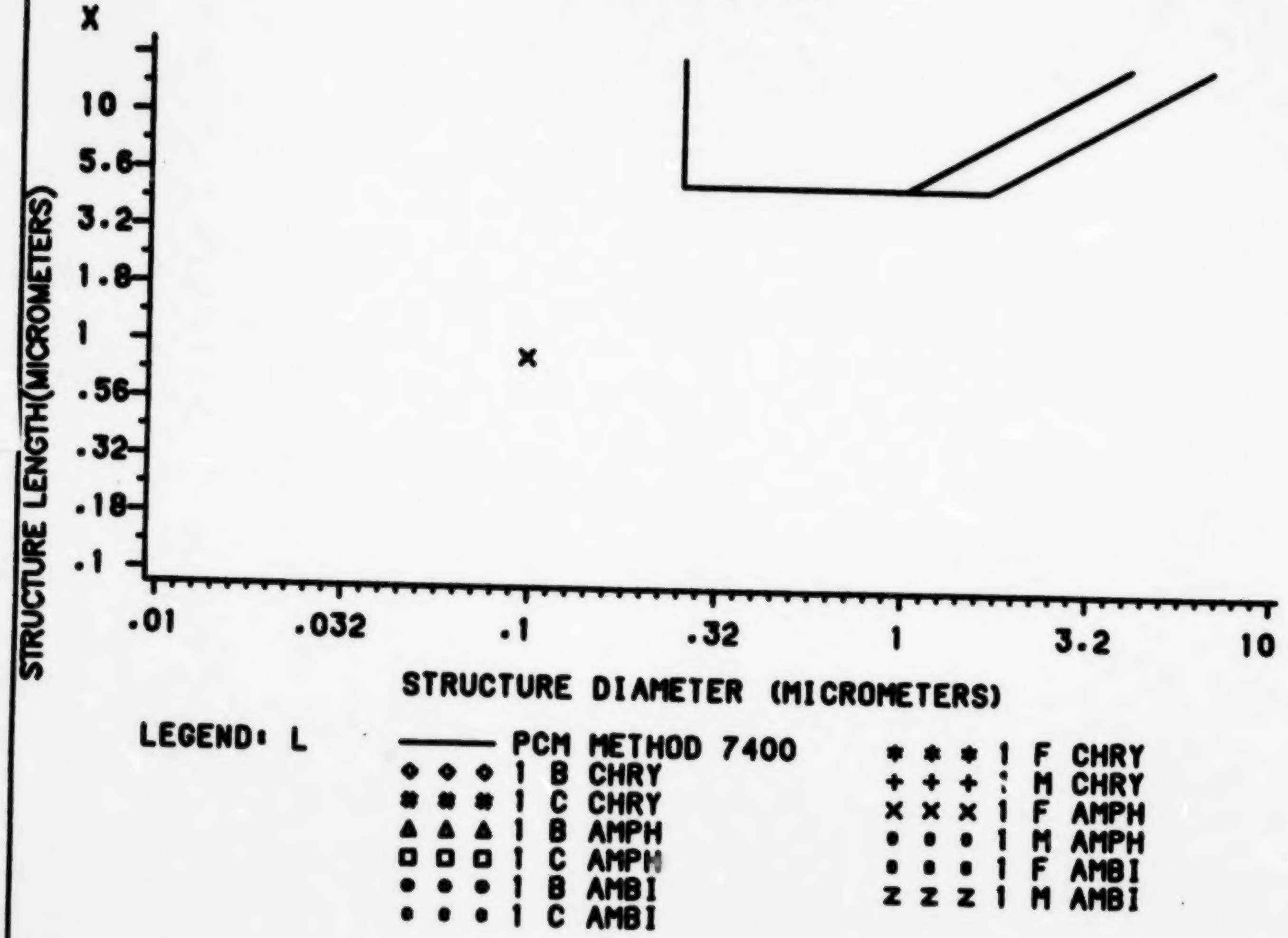
Distribution of structures in an Air Sample By TEM

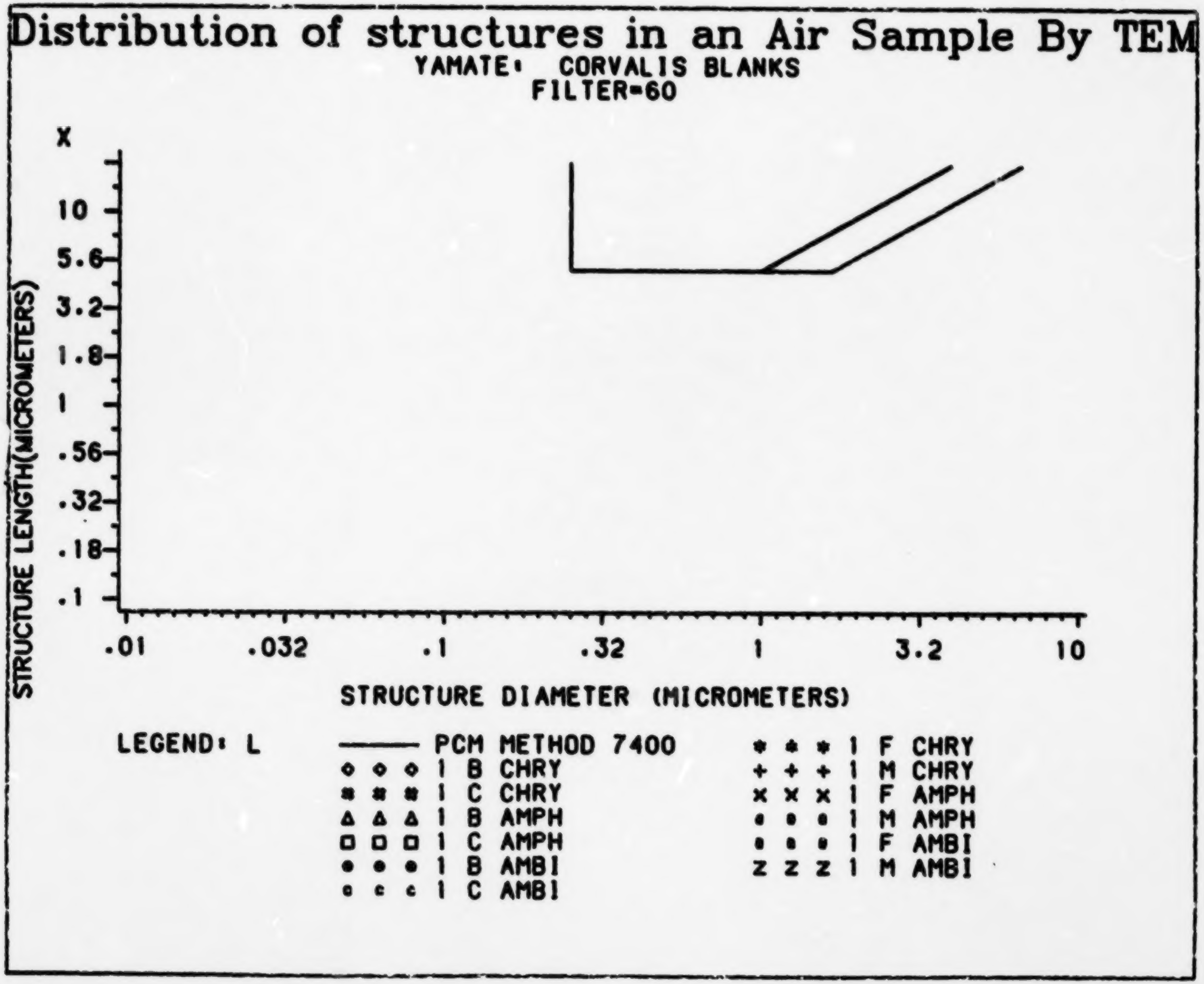
YAMATE: BLANKS
FILTER=106



Distribution of Structures in an Air Sample by TEM

MCCRONE: CORVALIS BLANKS
FILTER=60



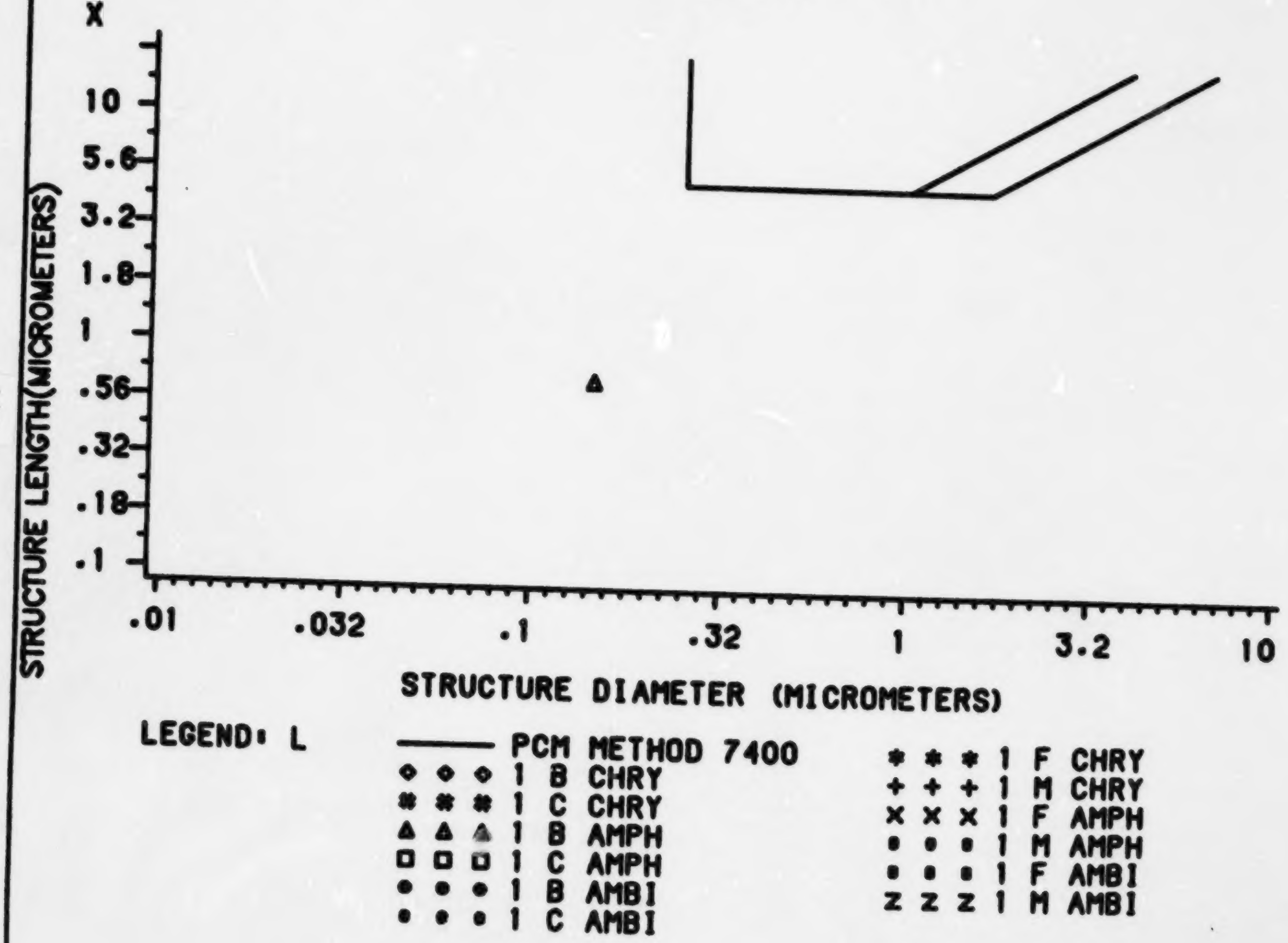


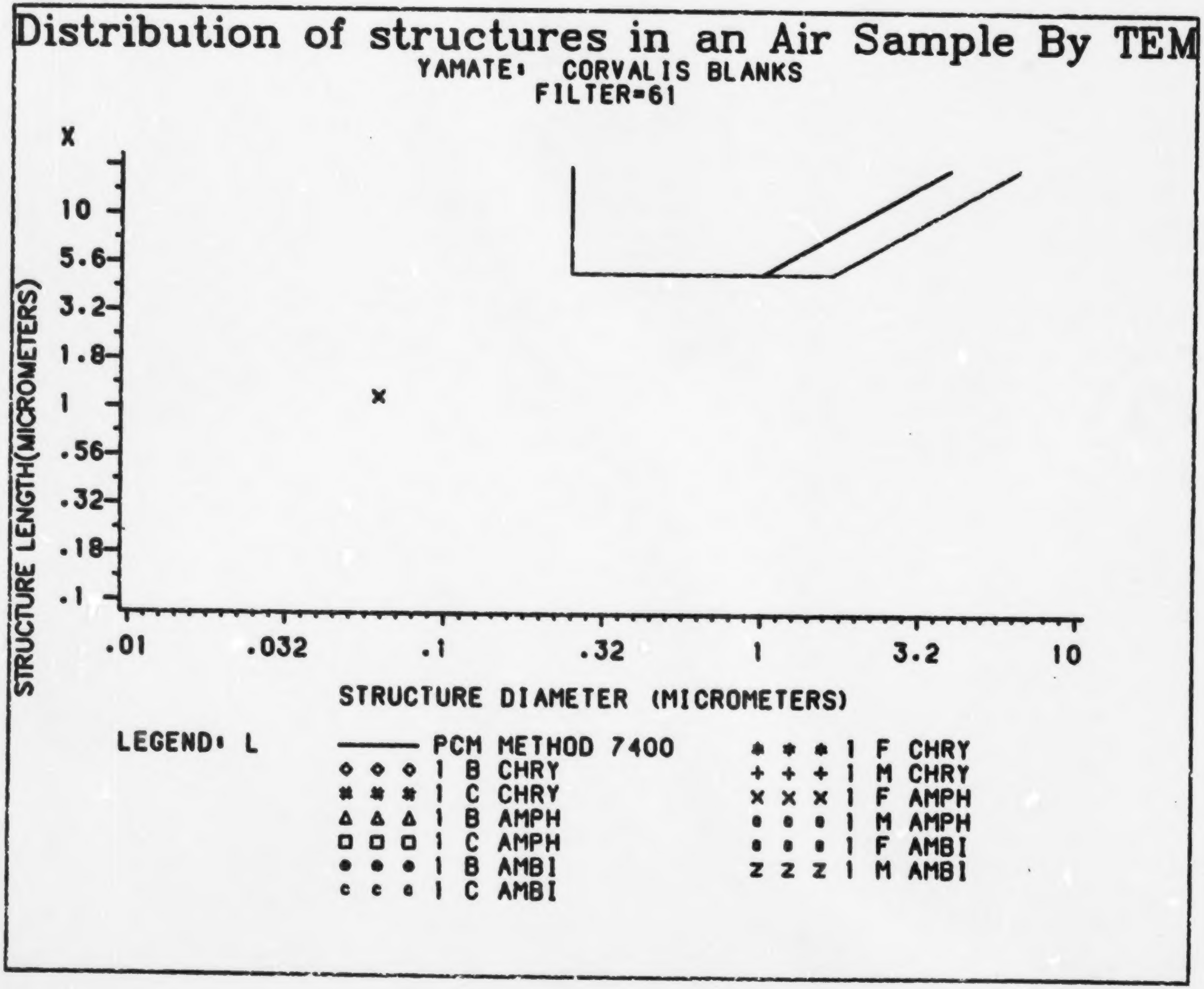
Distribution of structures in an Air Sample By TEM

YAMATE: CORVALIS BLANKS
FILTER=60

Distribution of Structures in an Air Sample by TEM

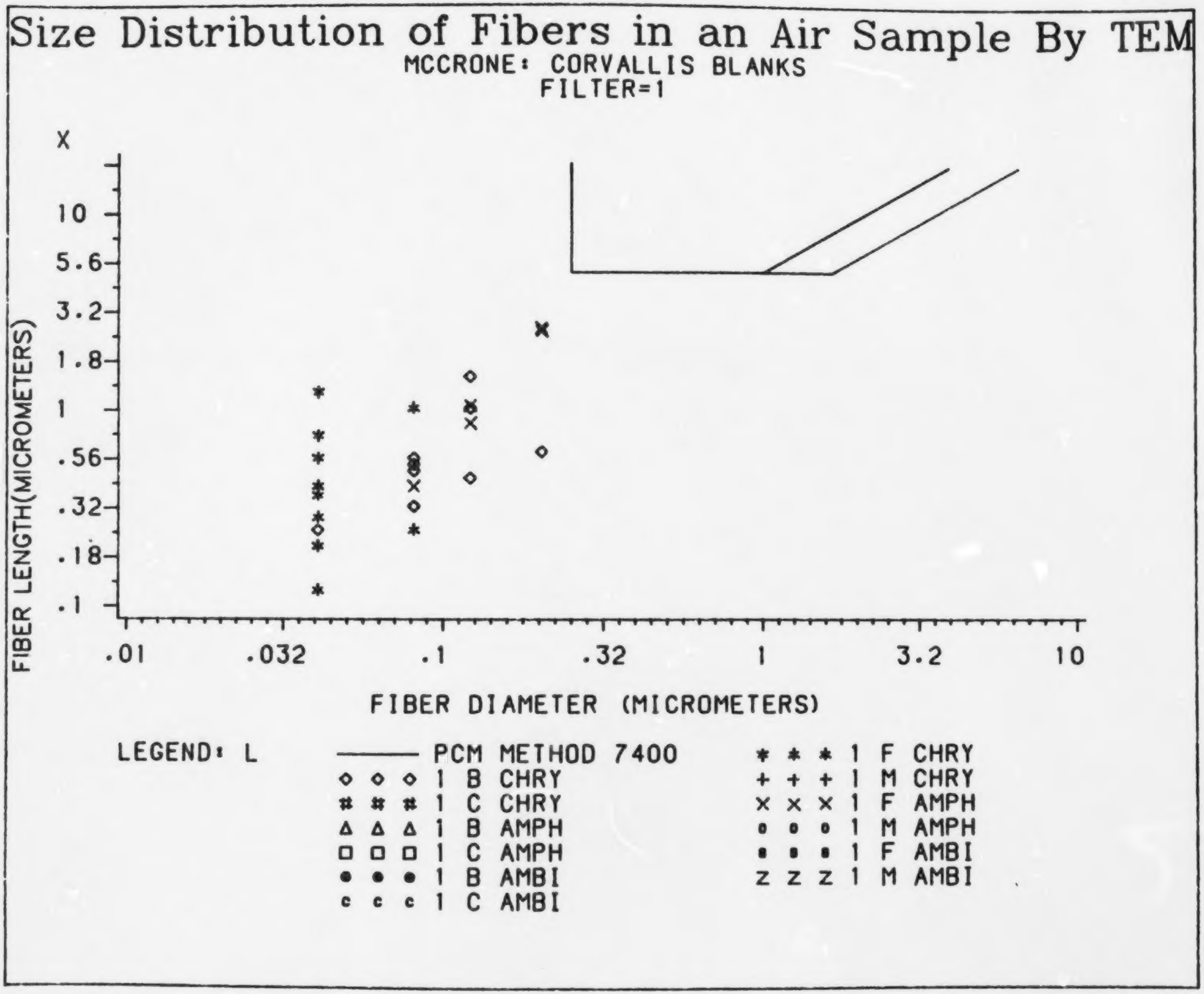
MCCRONE: CORVALIS BLANKS
FILTER=61




Distribution of structures in an Air Sample By TEM

YAMATE: CORVALIS BLANKS

FILTER=61

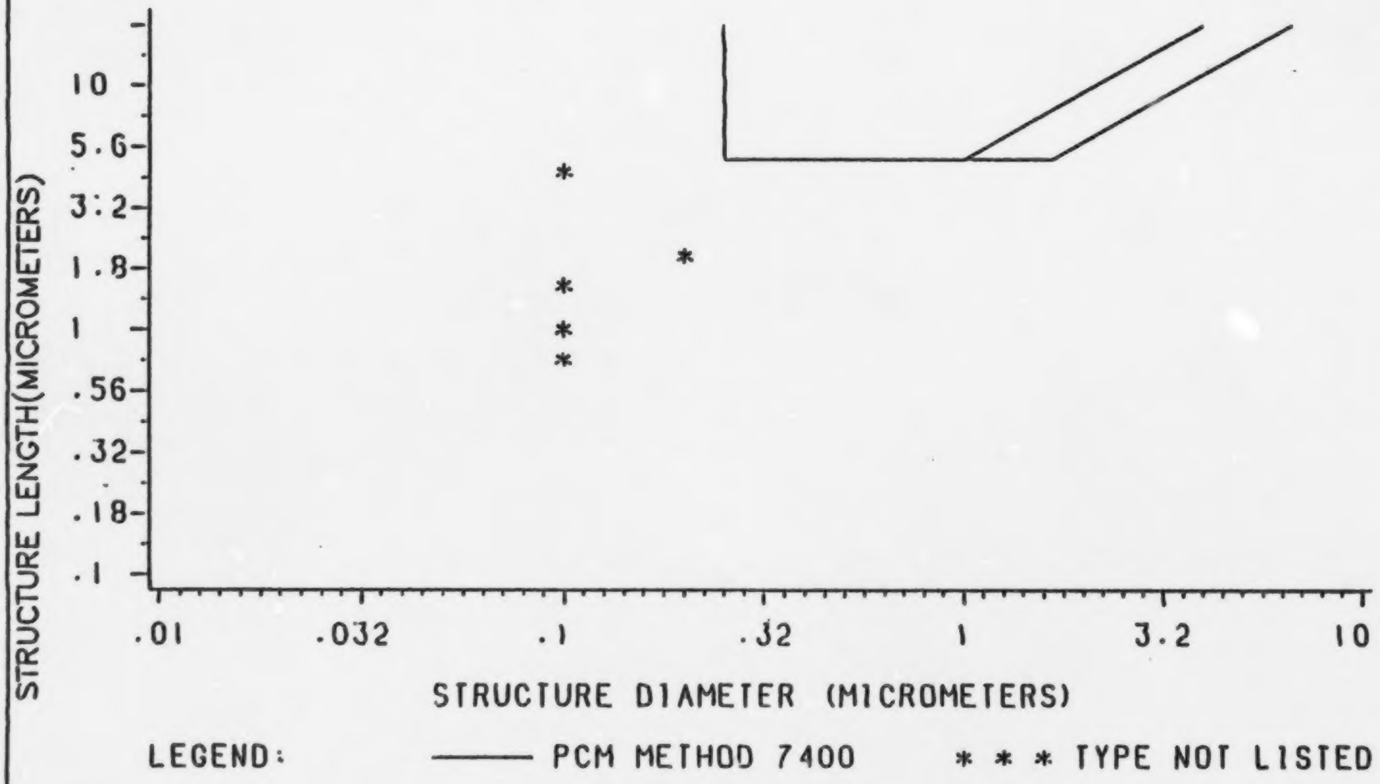


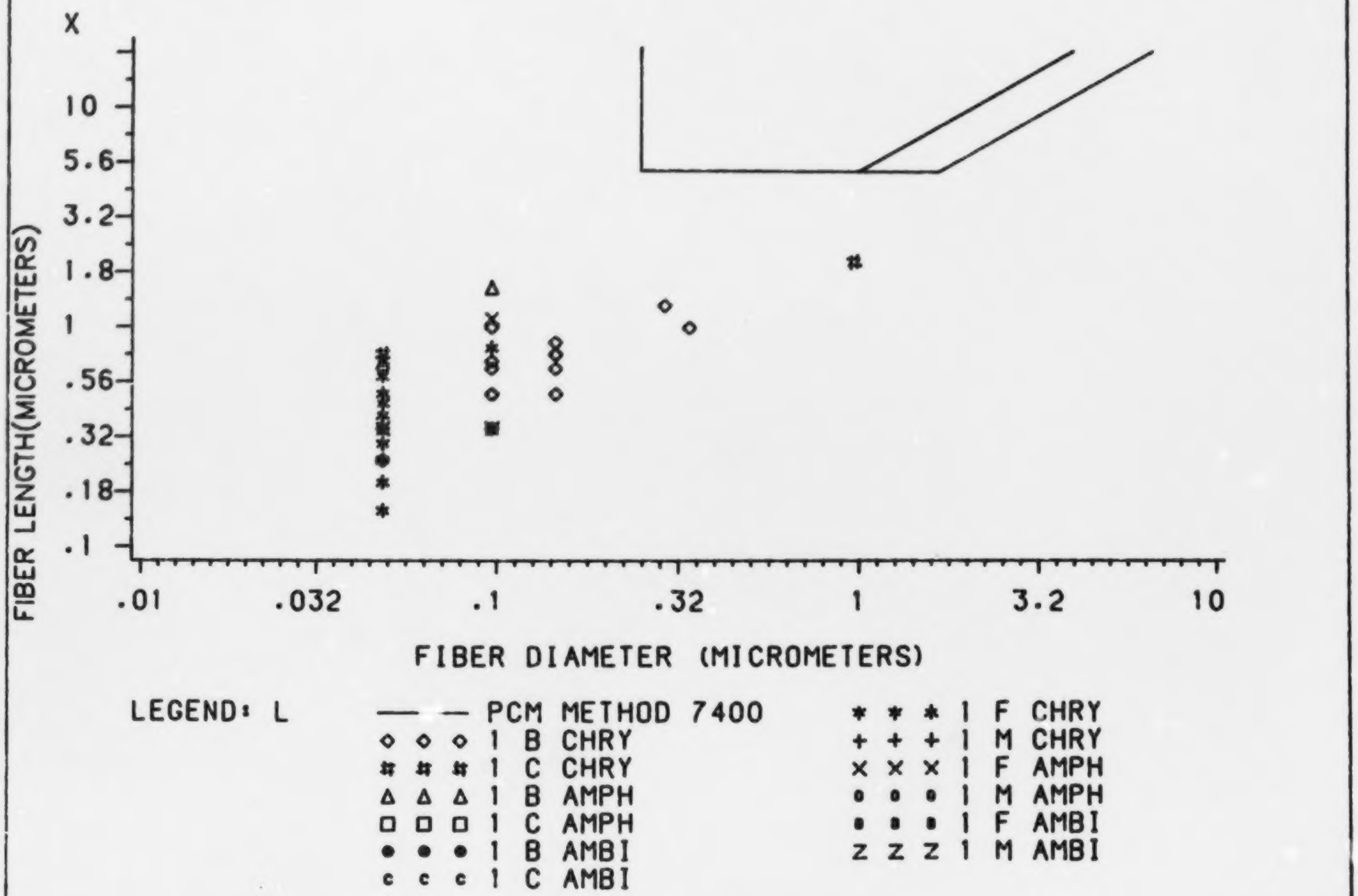
Size Distribution of Fibers in an Air Sample By TEM

MCCRONE: CORVALLIS BLANKS
FILTER=1

Distribution of Structures in an Air Sample by TEM

NIOSH: CORVALLIS BLANKS. 169 GRID OPENINGS COUNTED
FILTER=1

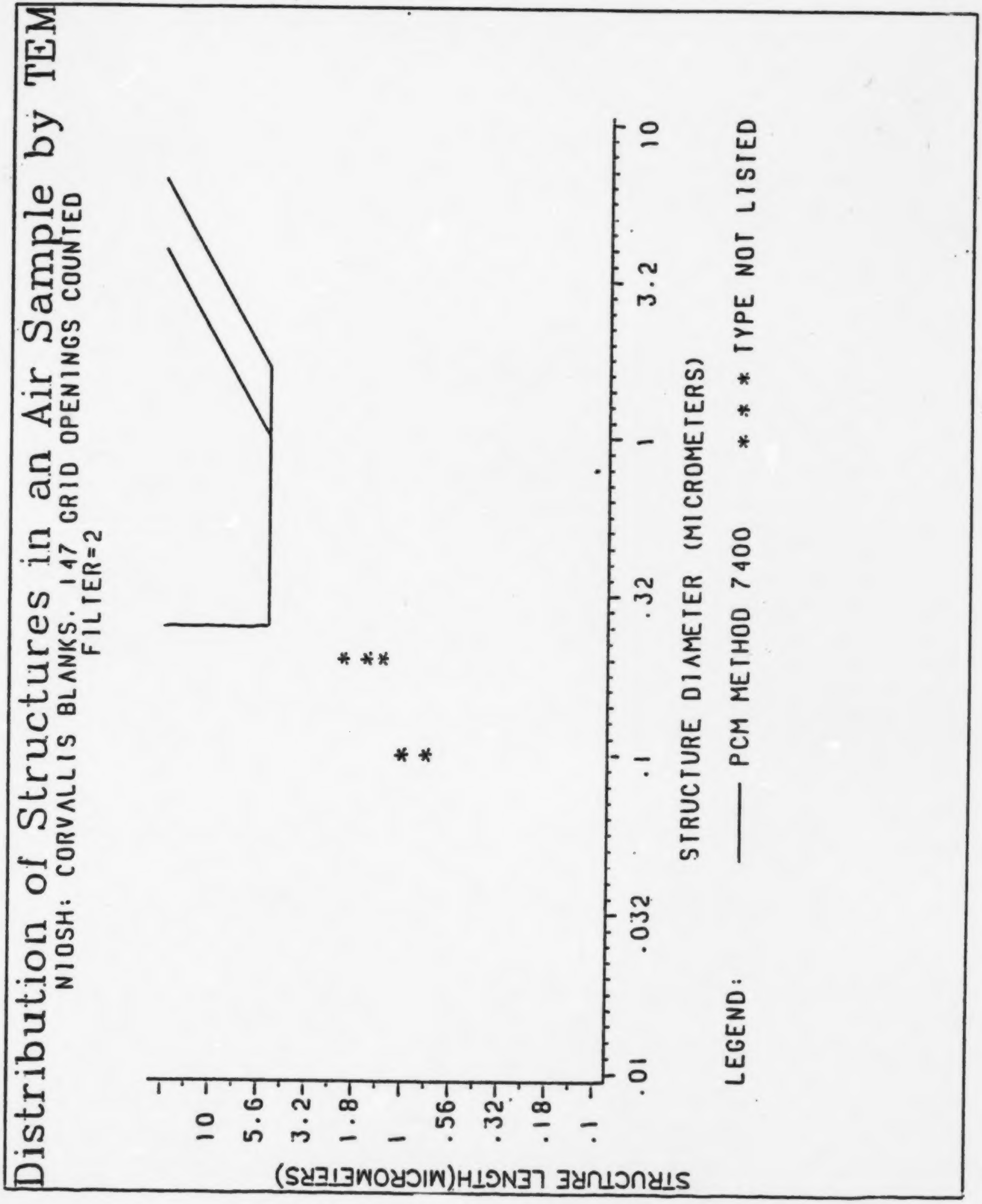




Size Distribution of Fibers in an Air Sample By TEM

MCCRONE: CORVALLIS BLANKS

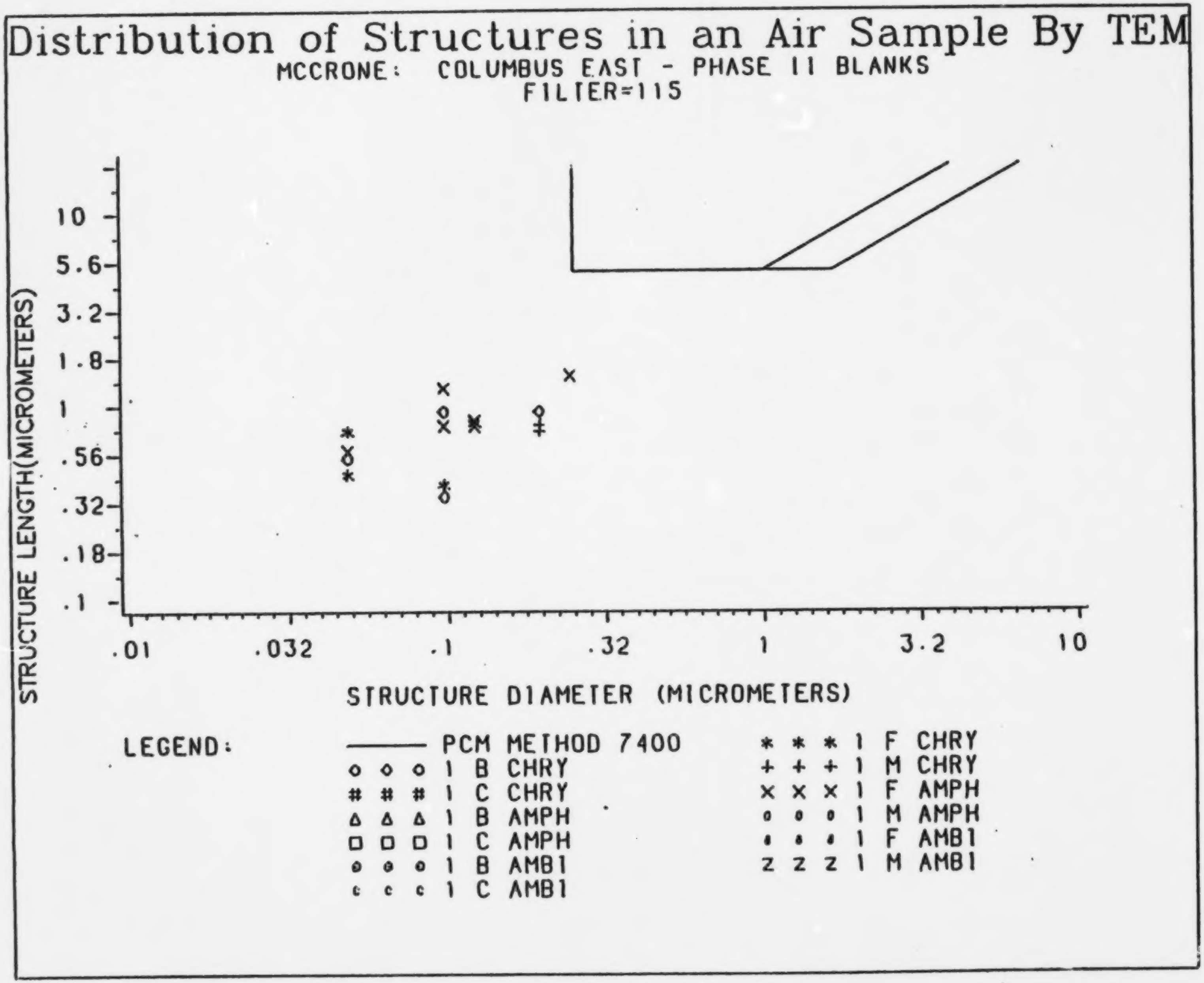
FILTER=2



Distribution of Structures in an Air Sample by TEM

NIOSH: CORVALLIS BLANKS. 147 GRID OPENINGS COUNTED

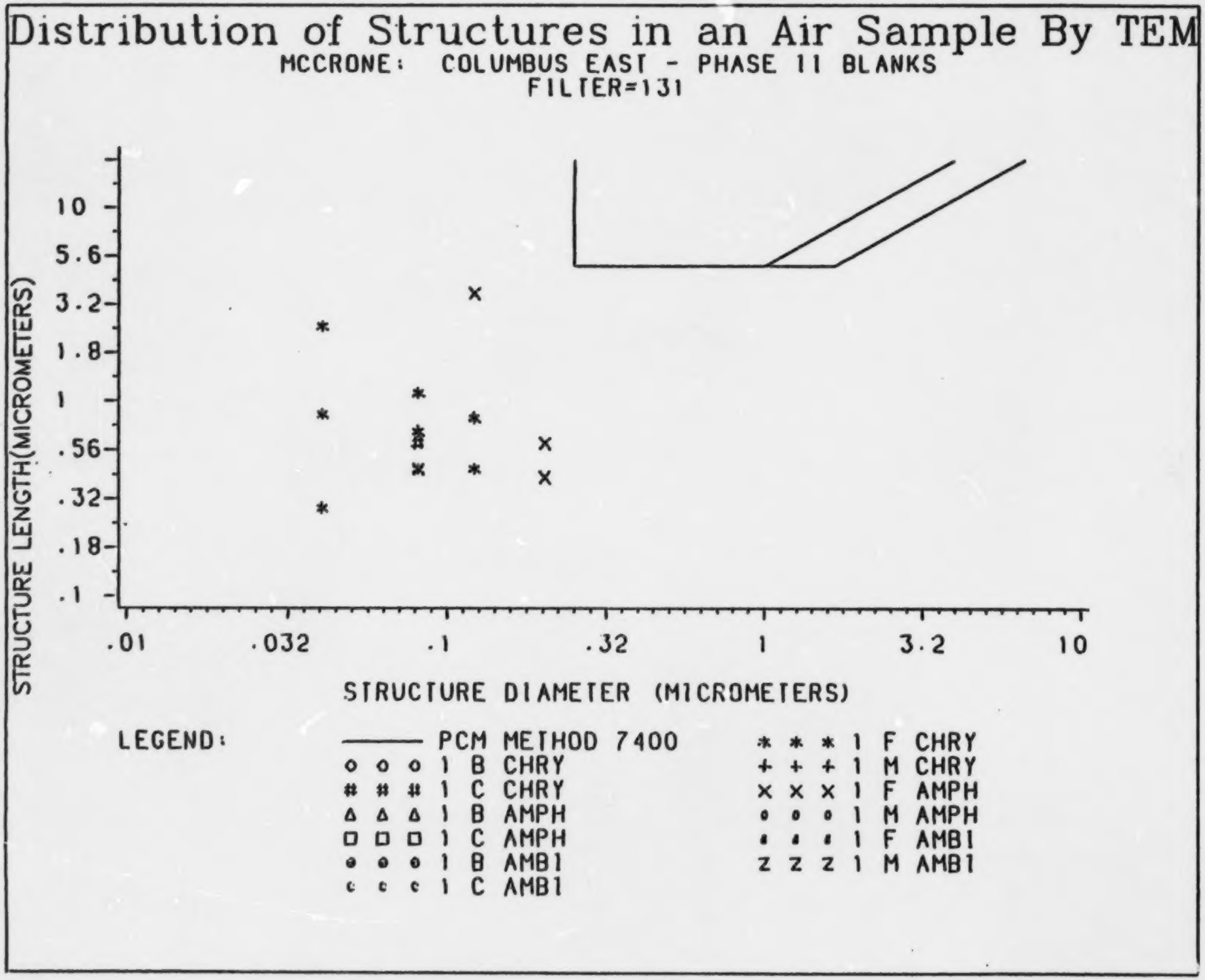
FILTER=2

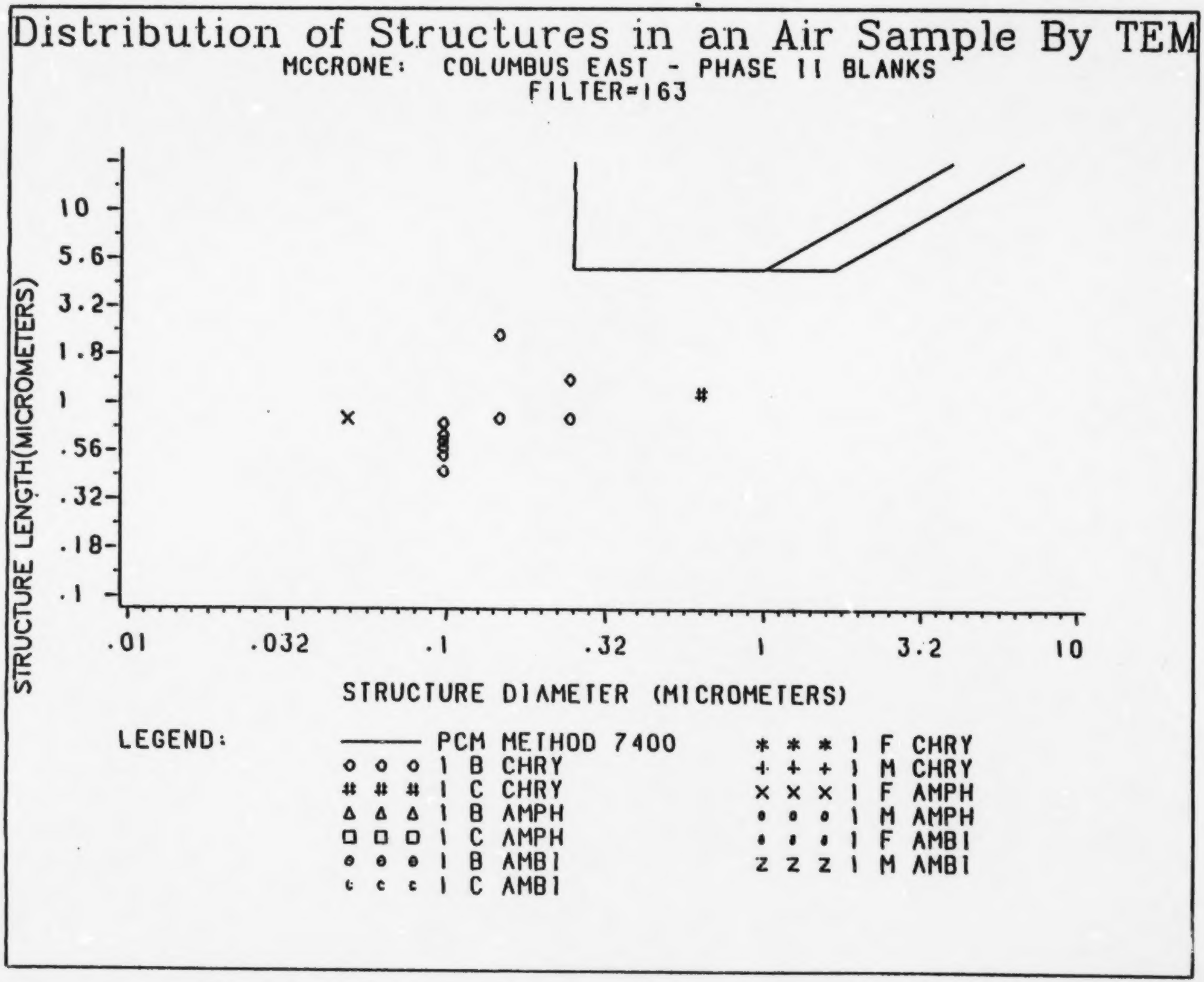


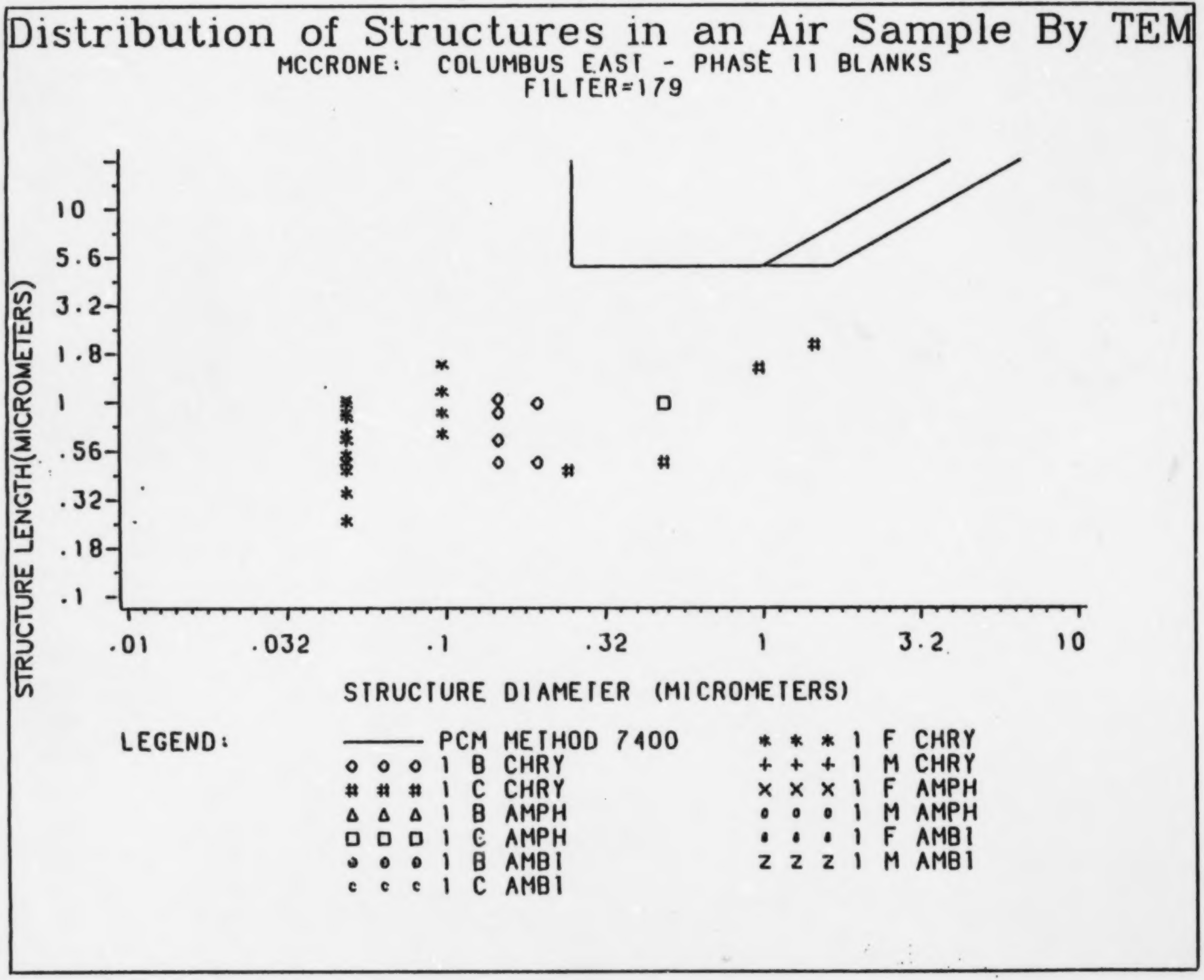
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS

FILTER=115


Distribution of Structures in an Air Sample By TEM
MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=131
10
5.6
3.2
1.8
1
.56
.32
.18
.1
STRUCTURE LENGTH(MICROMETERS)
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND:
——— PCM METHOD 7400
○ ○ ○ 1 B CHRY
# 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
c c c 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
• • • 1 M AMPH
▪ ▪ ▪ 1 F AMBI
z z z 1 M AMBI


Distribution of Structures in an Air Sample By TEM
MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=163
STRUCTURE LENGTH(MICROMETERS)
10
5.6
3.2
1.8
1
.56
.32
.18
.1
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND:
——— PCM METHOD 7400
o o o 1 B CHRY
# 1 C CHRY
Δ Δ Δ 1 B AMPH
□ □ □ 1 C AMPH
ɵ ɵ ɵ 1 B AMBI
c c c 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
• • • 1 M AMPH
• • • 1 F AMBI
Z Z Z 1 M AMBI

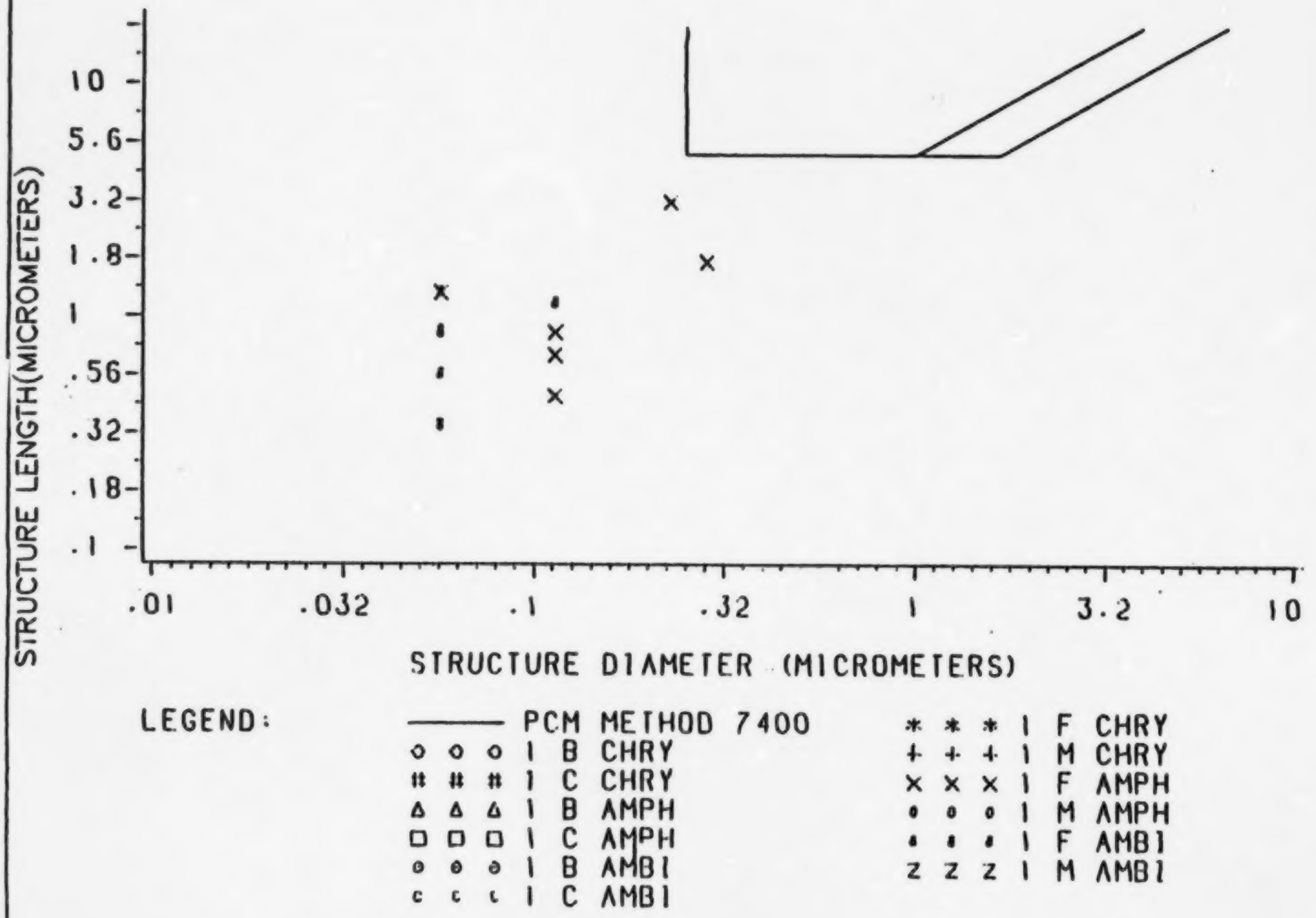


Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS
FILTER=179

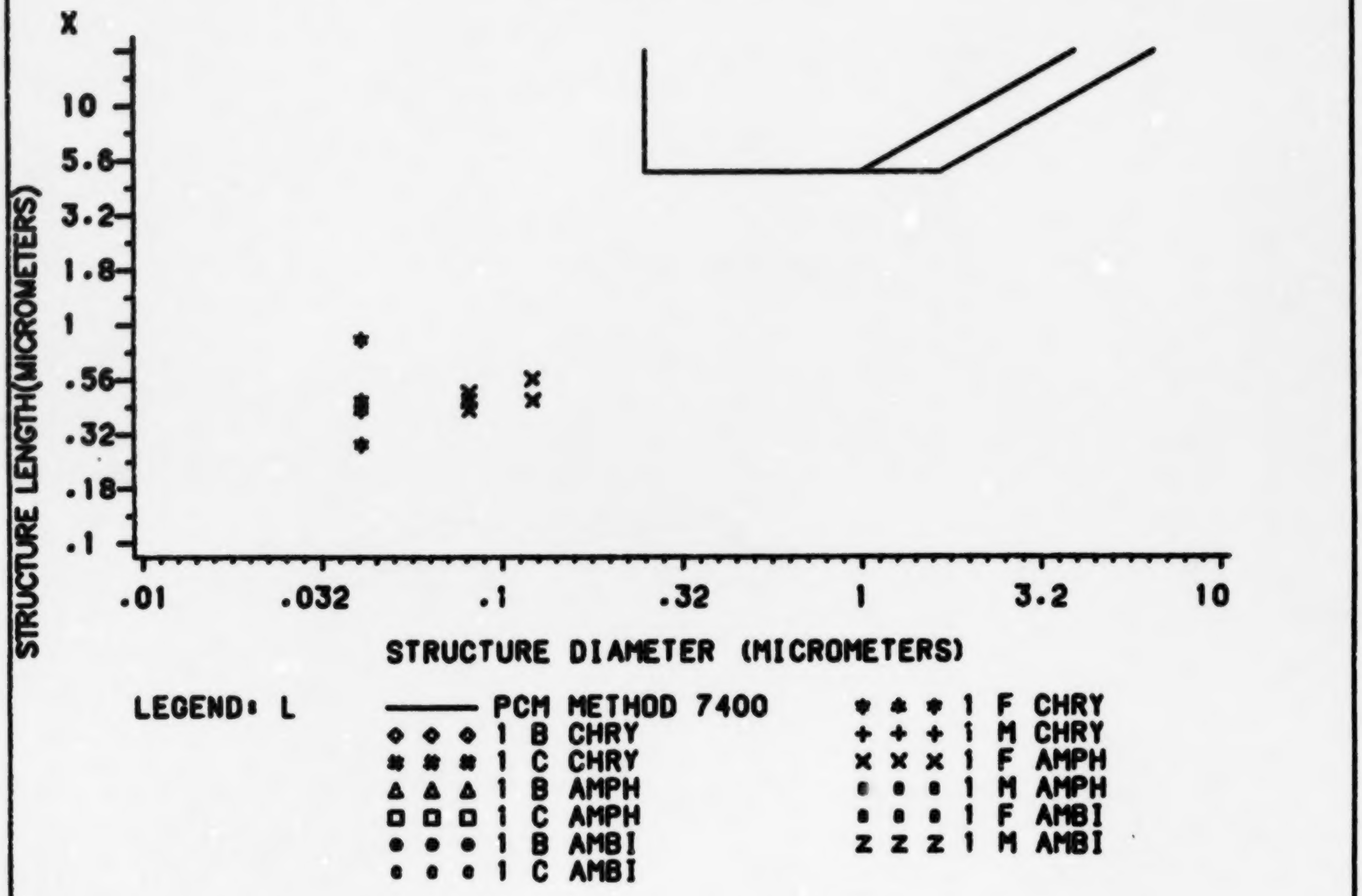
Distribution of Structures in an Air Sample by TEM

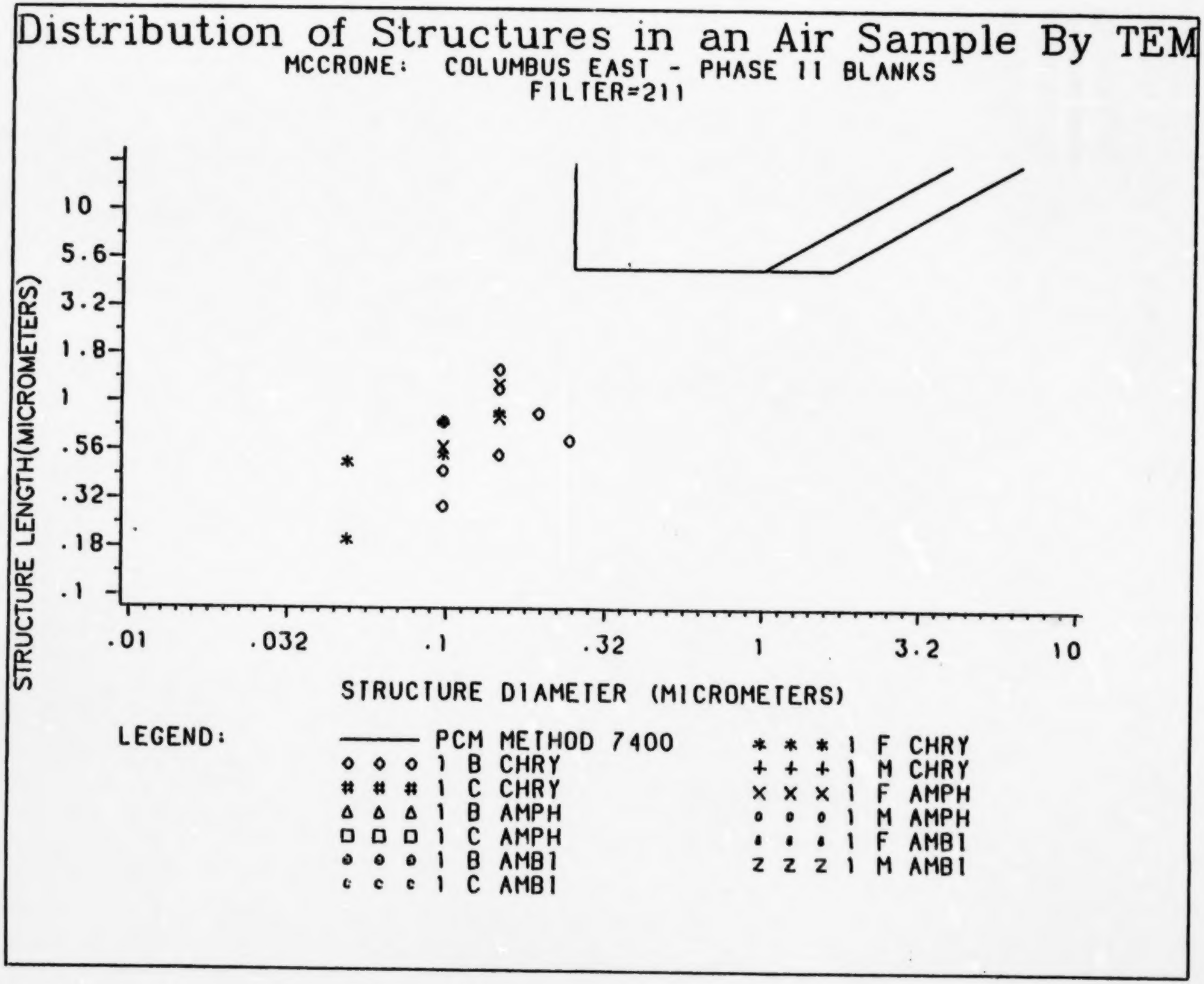
NIOSH: DUPLICATE ANALYSIS OF MCCRONE BLANKS
FILTER=179



Distribution of Structures in an Air Sample By TEM

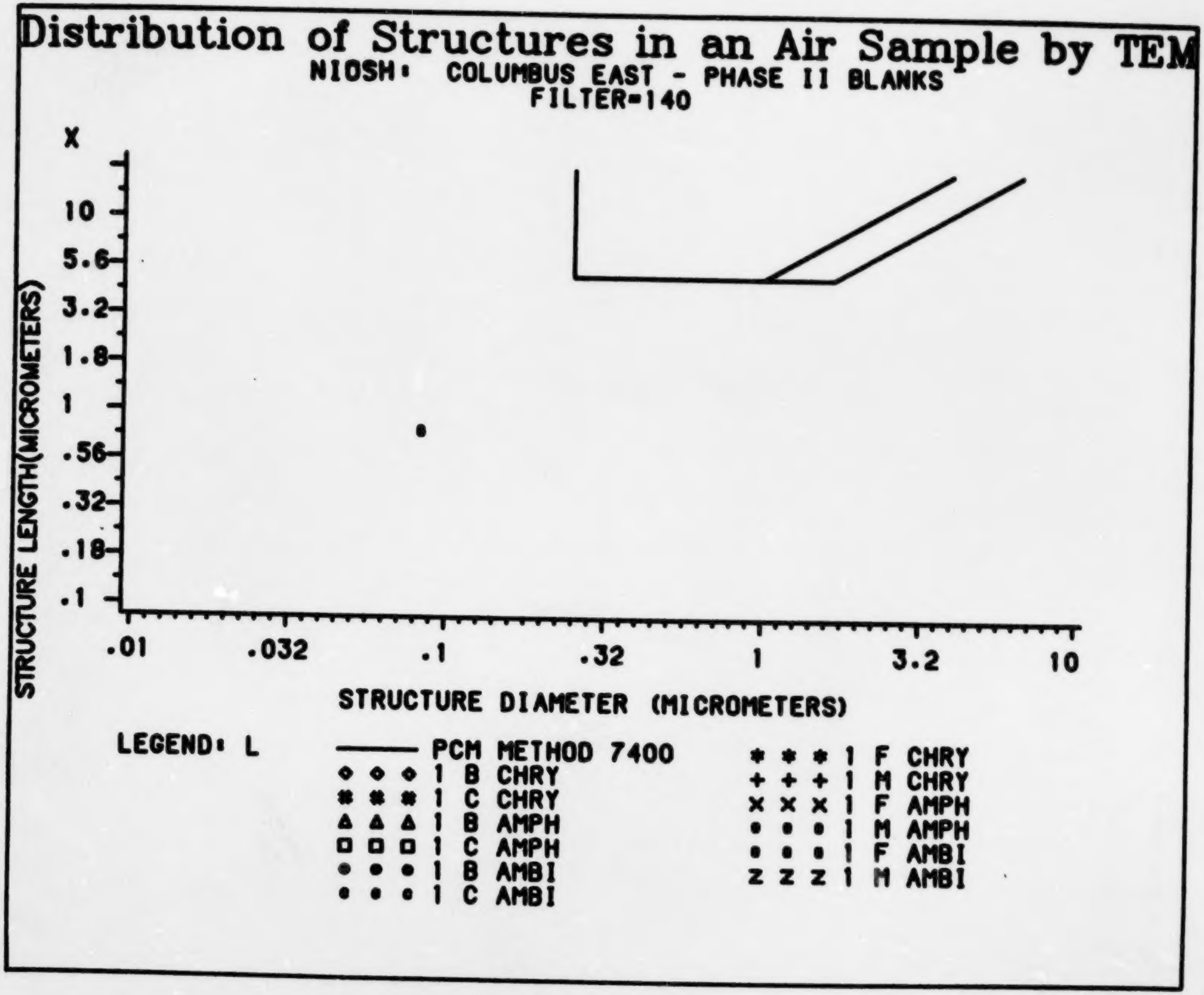
MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=195

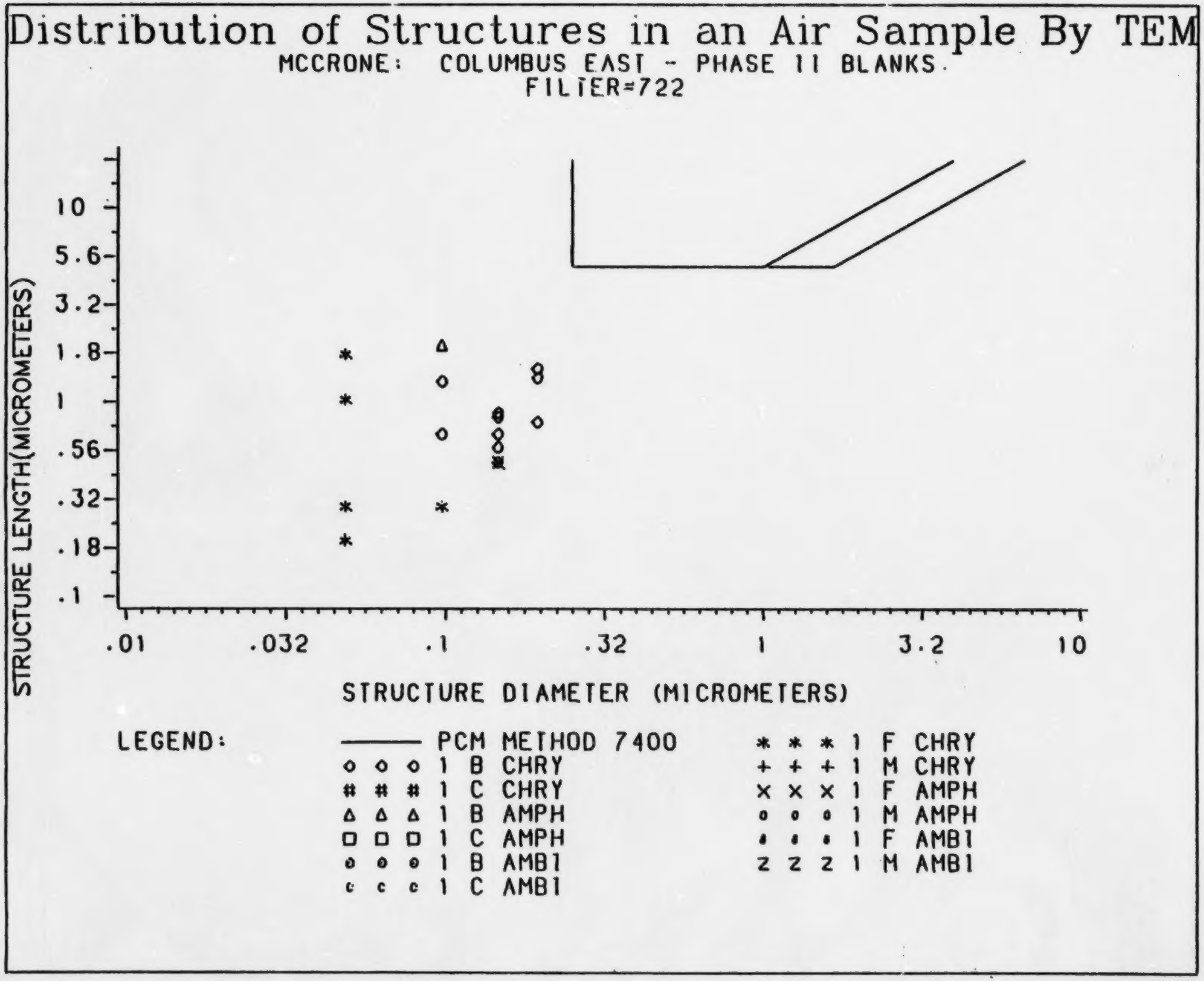




Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=211


Distribution of Structures in an Air Sample by TEM
NIOSH: COLUMBUS EAST - PHASE II BLANKS
FILTER=140
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
STRUCTURE LENGTH(MICROMETERS)
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
PCM METHOD 7400
◇ ◇ ◇ 1 B CHRY
* * * 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
• • • 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
• • • 1 M AMPH
• • • 1 F AMBI
Z Z Z 1 M AMBI



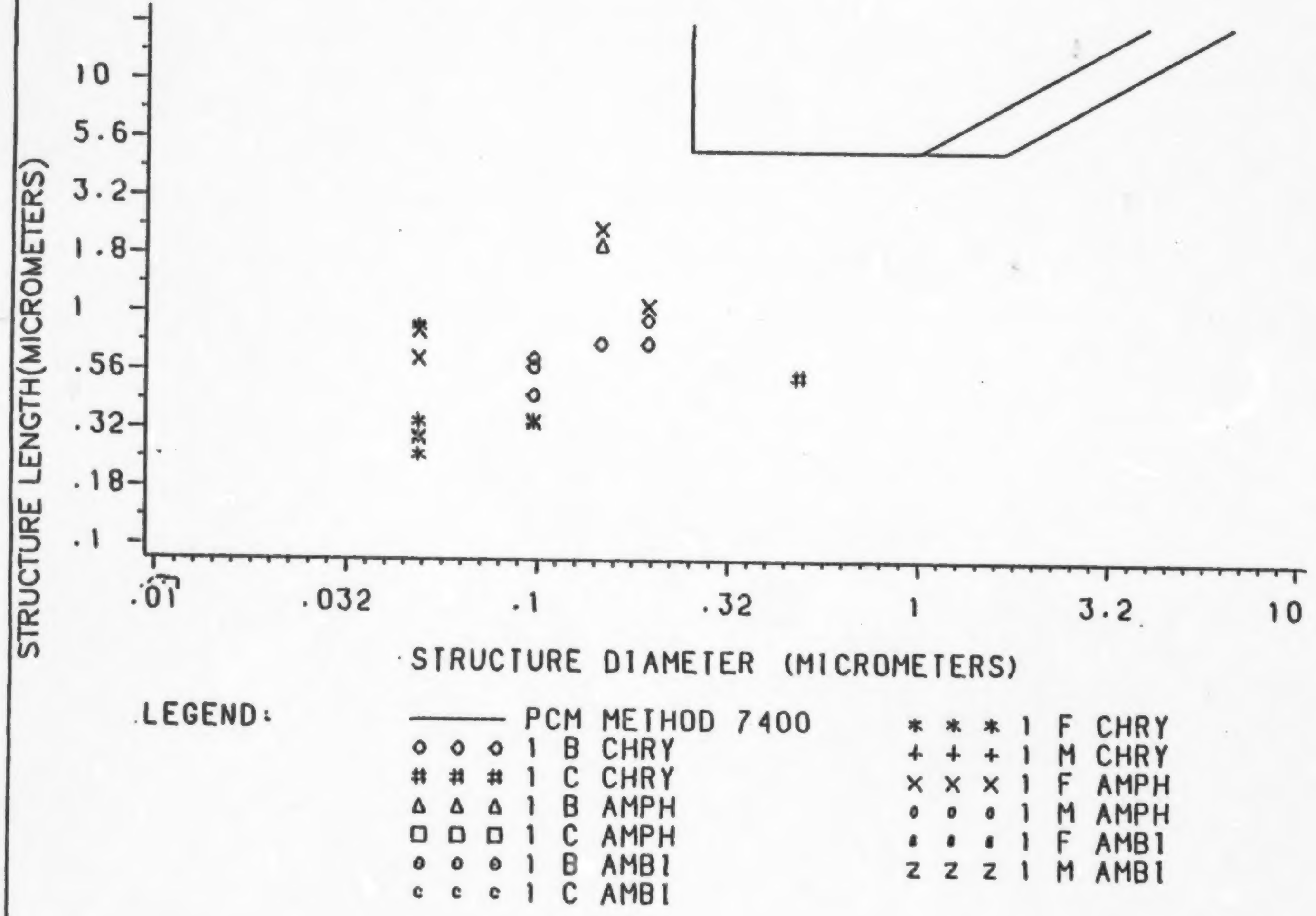
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS

FILTER=722

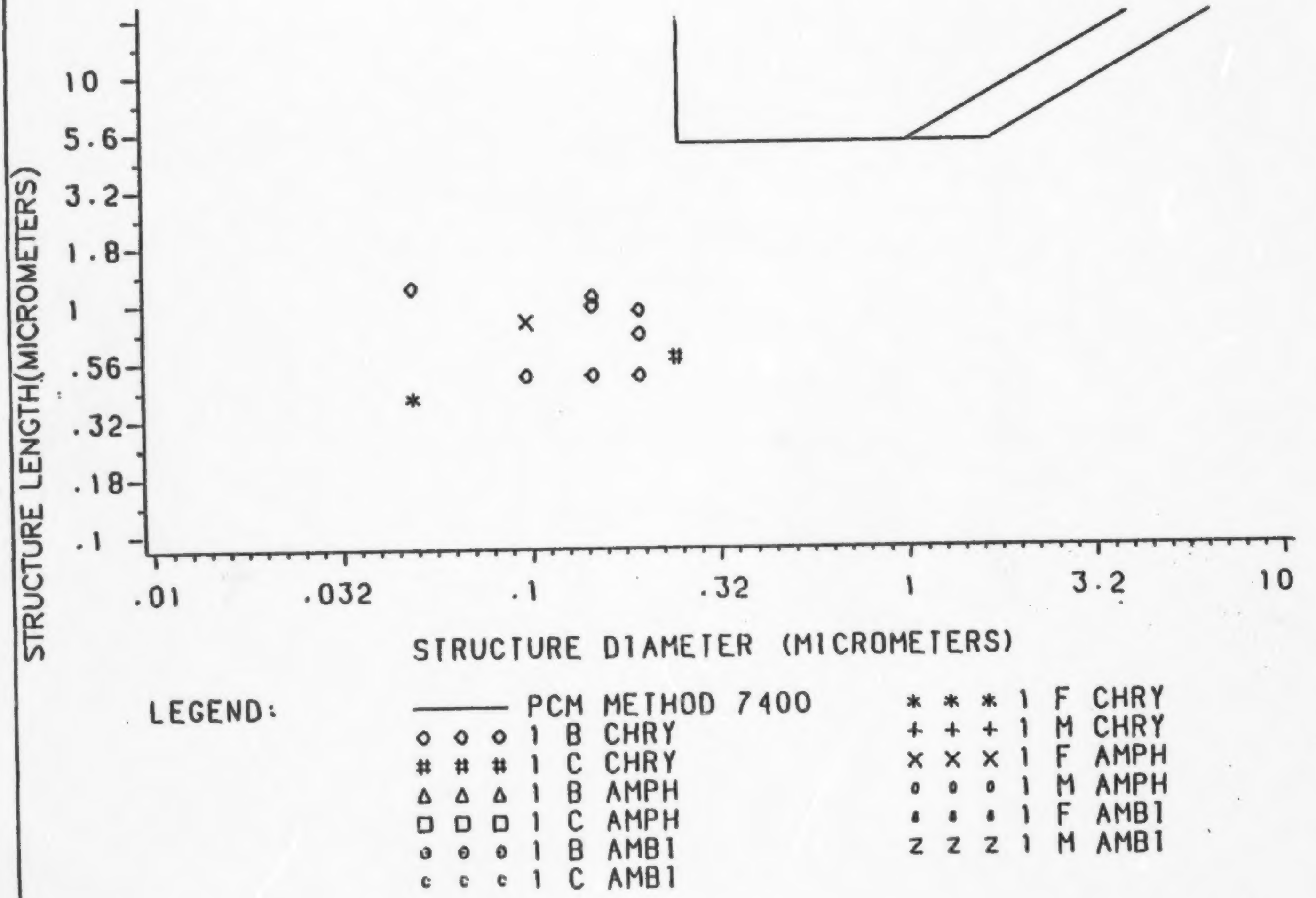
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=724



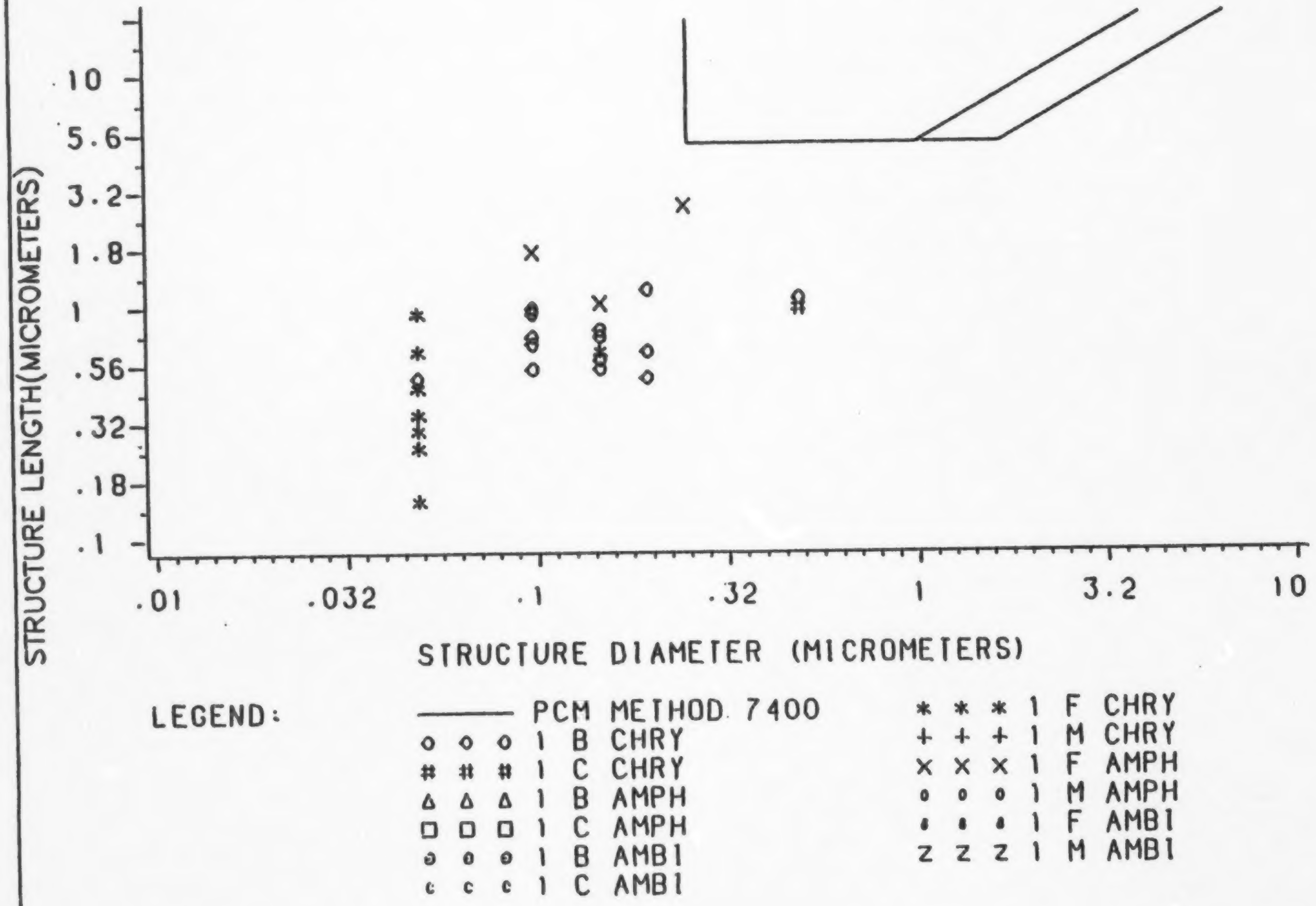
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=764



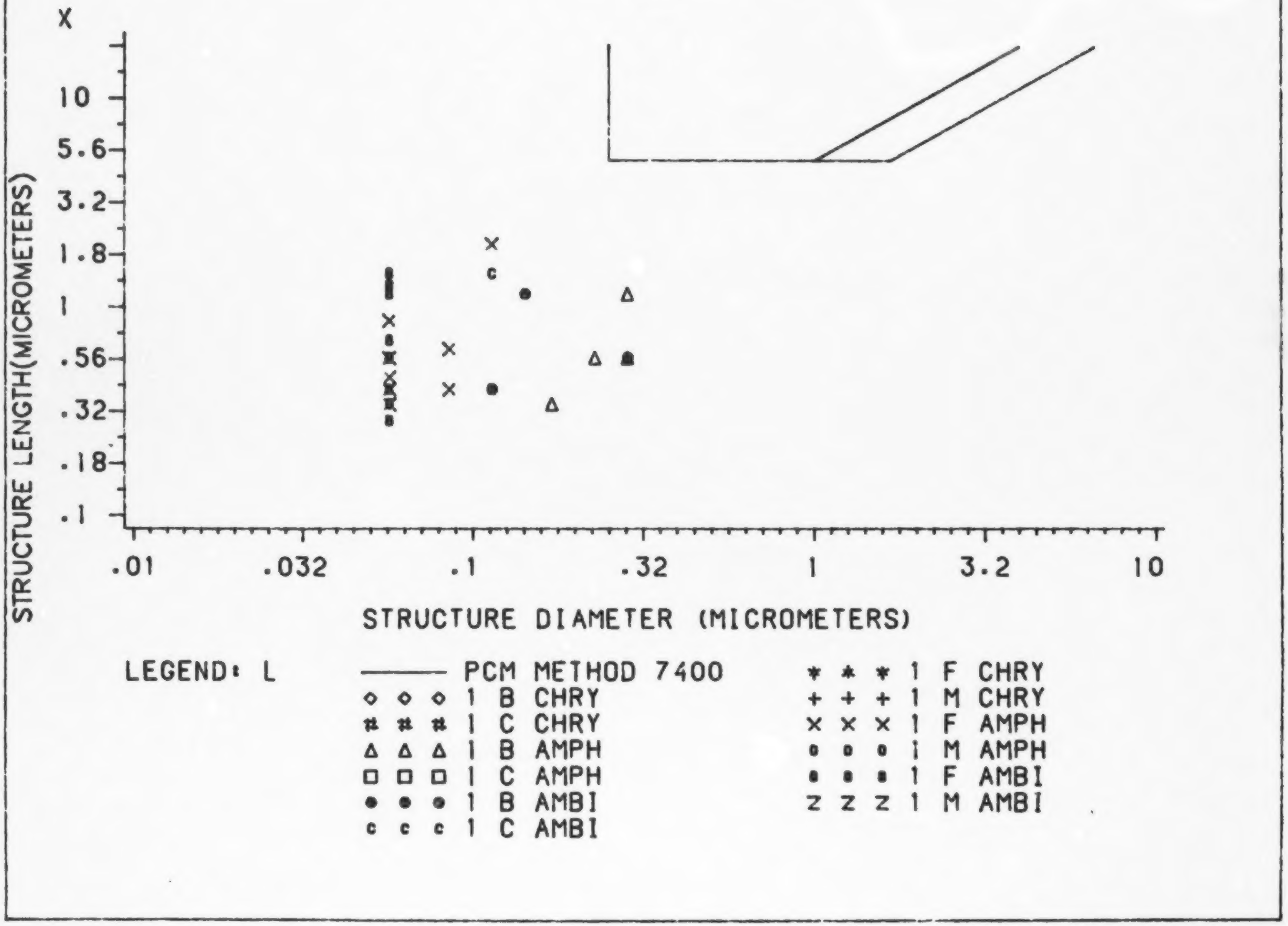
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS
FILTER=778



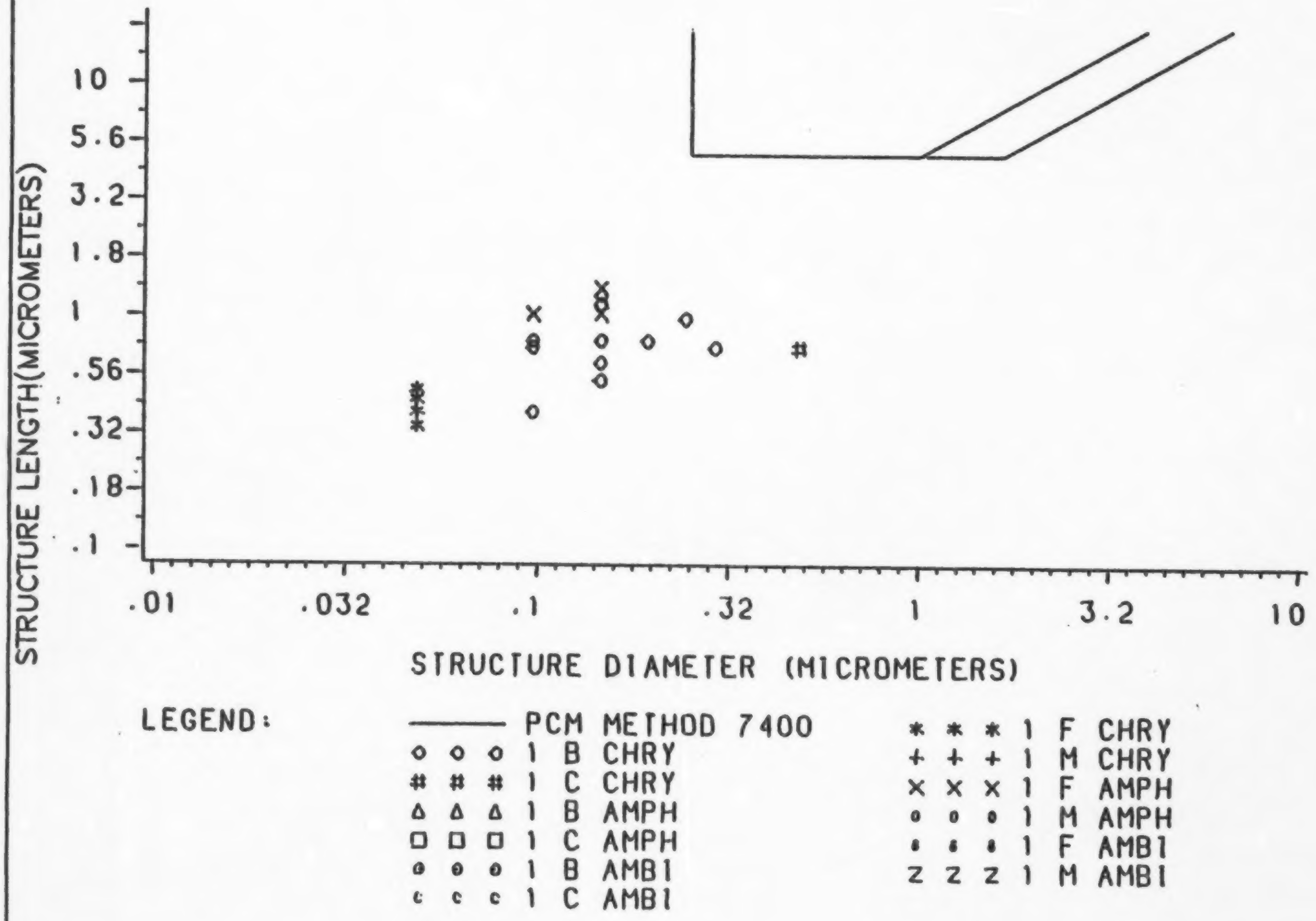
Distribution of Structures in an Air Sample by TEM

DUPLICATE ANALYSIS OF MCCRONE BLANKS
FILTER=778



Distribution of Structures in an Air Sample By TEM

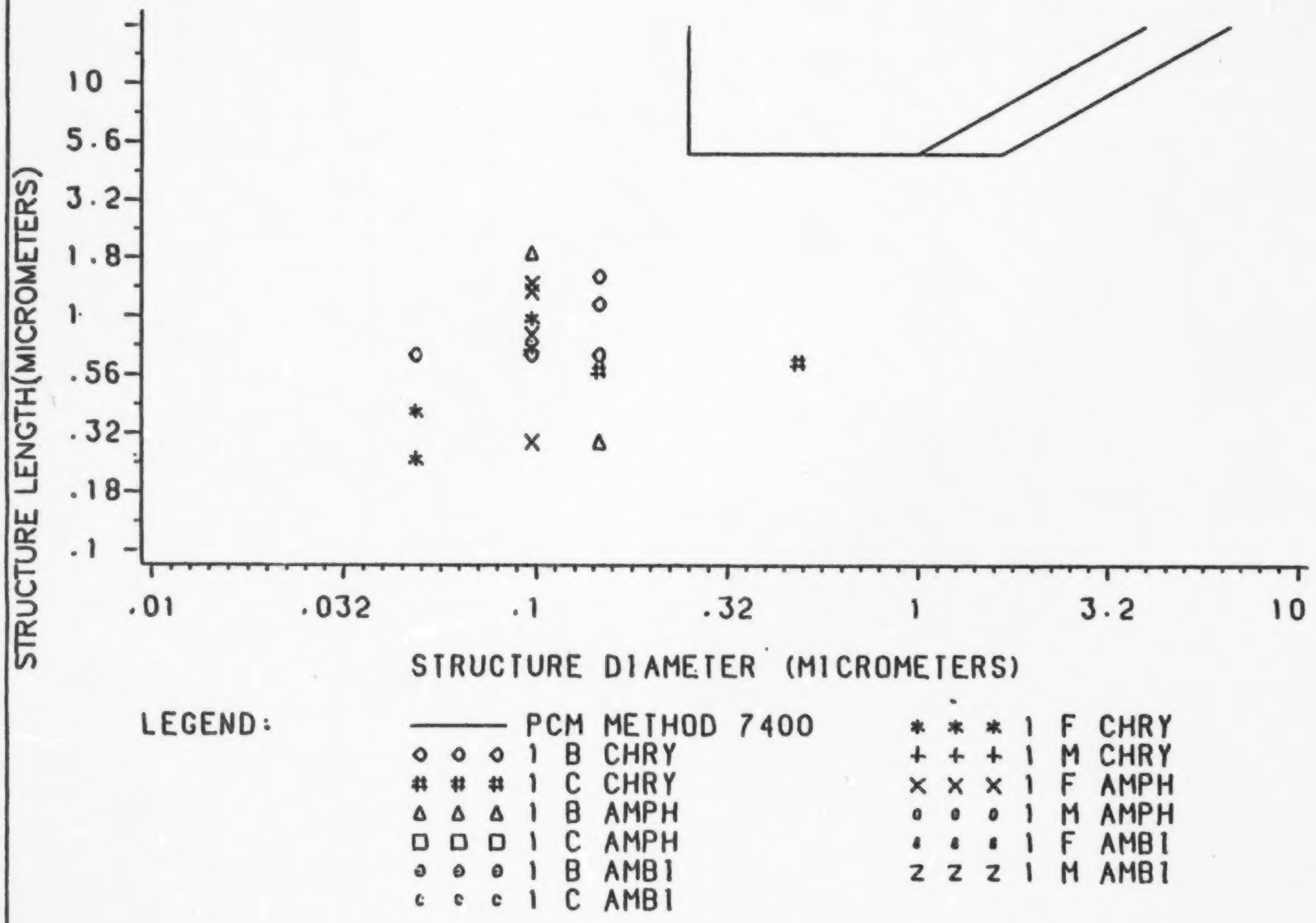
MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=984



Distribution of Structures in an Air Sample By TEM

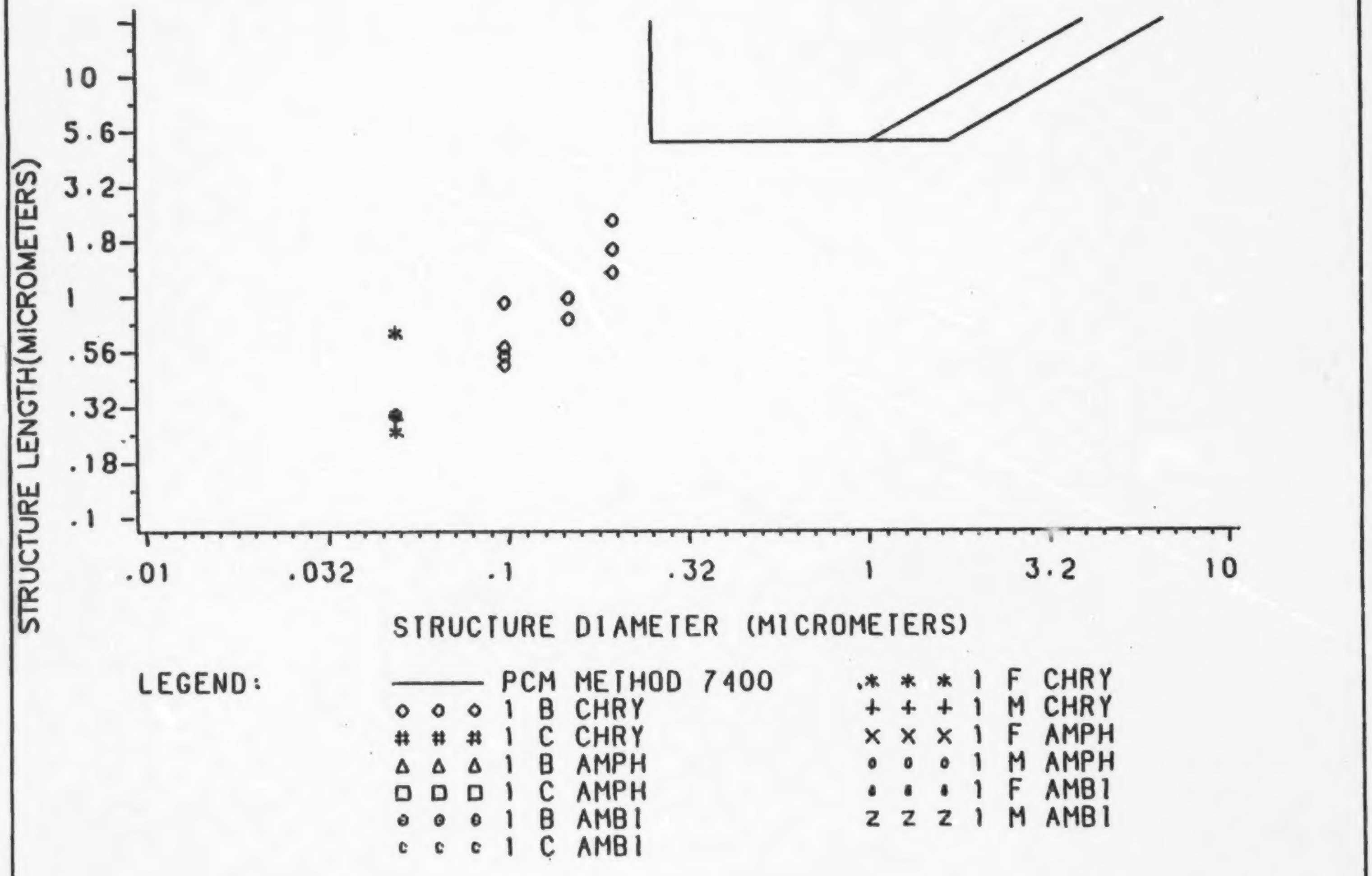
MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS

FILTER=1003



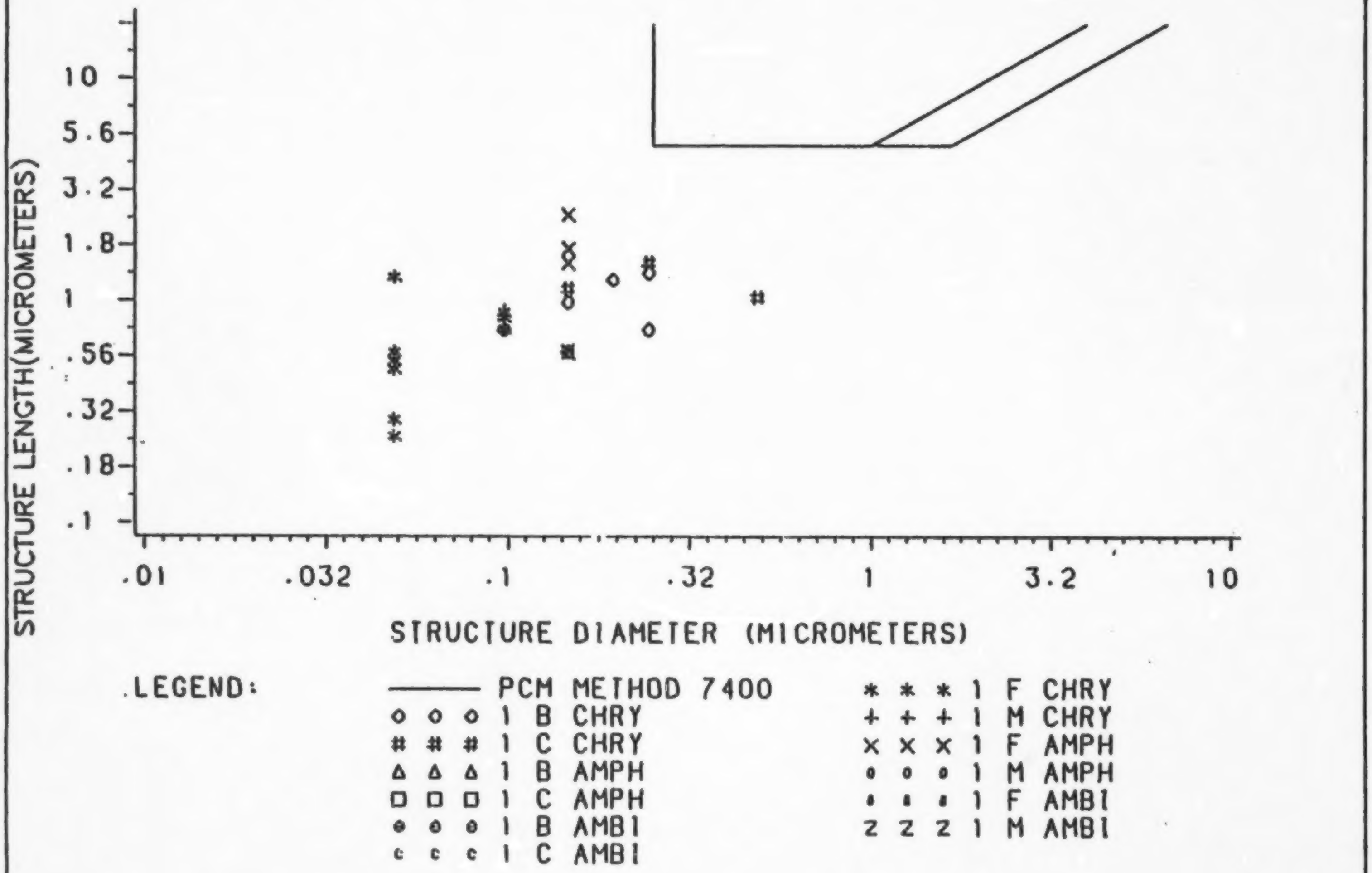
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=1099



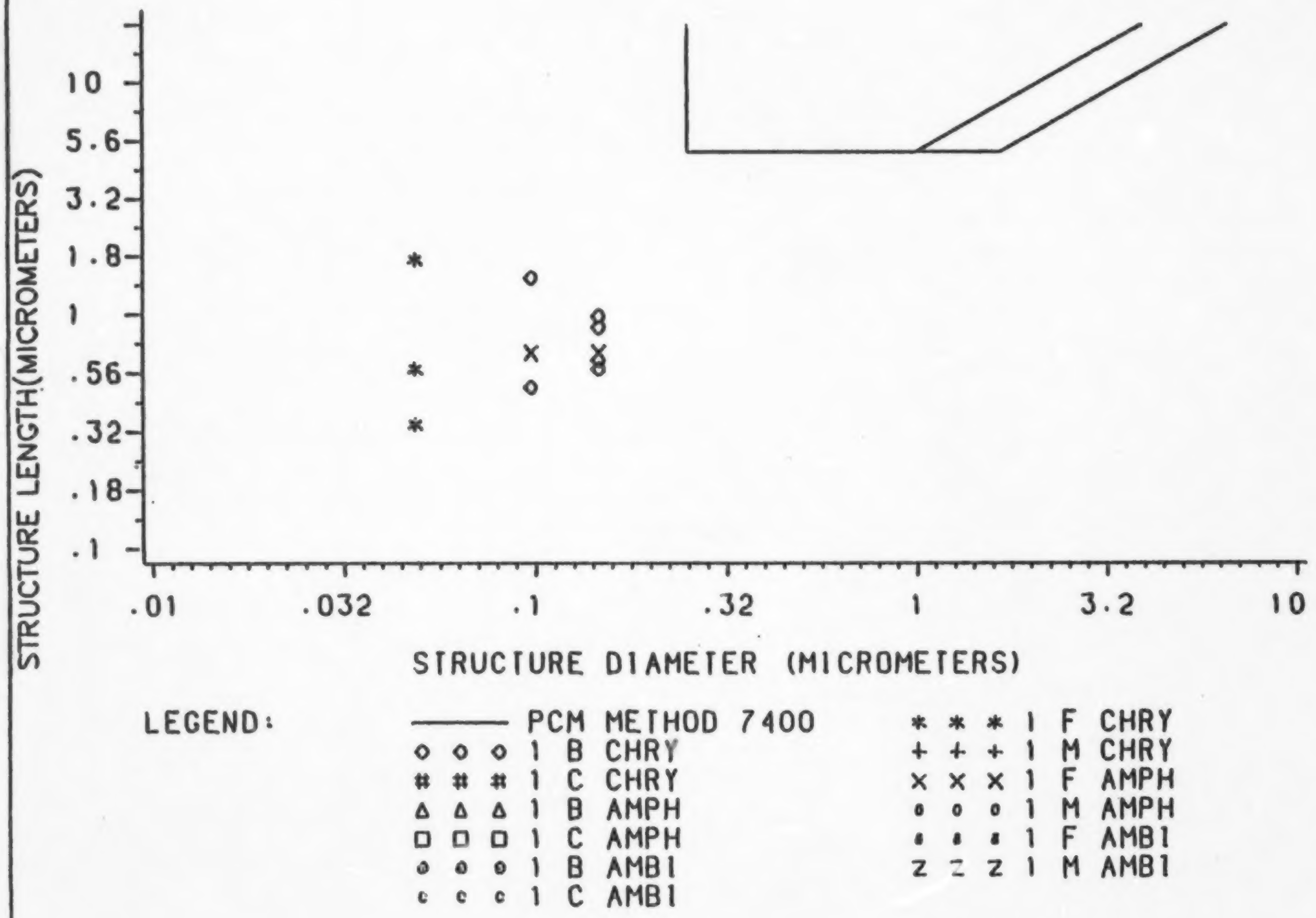
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=1109



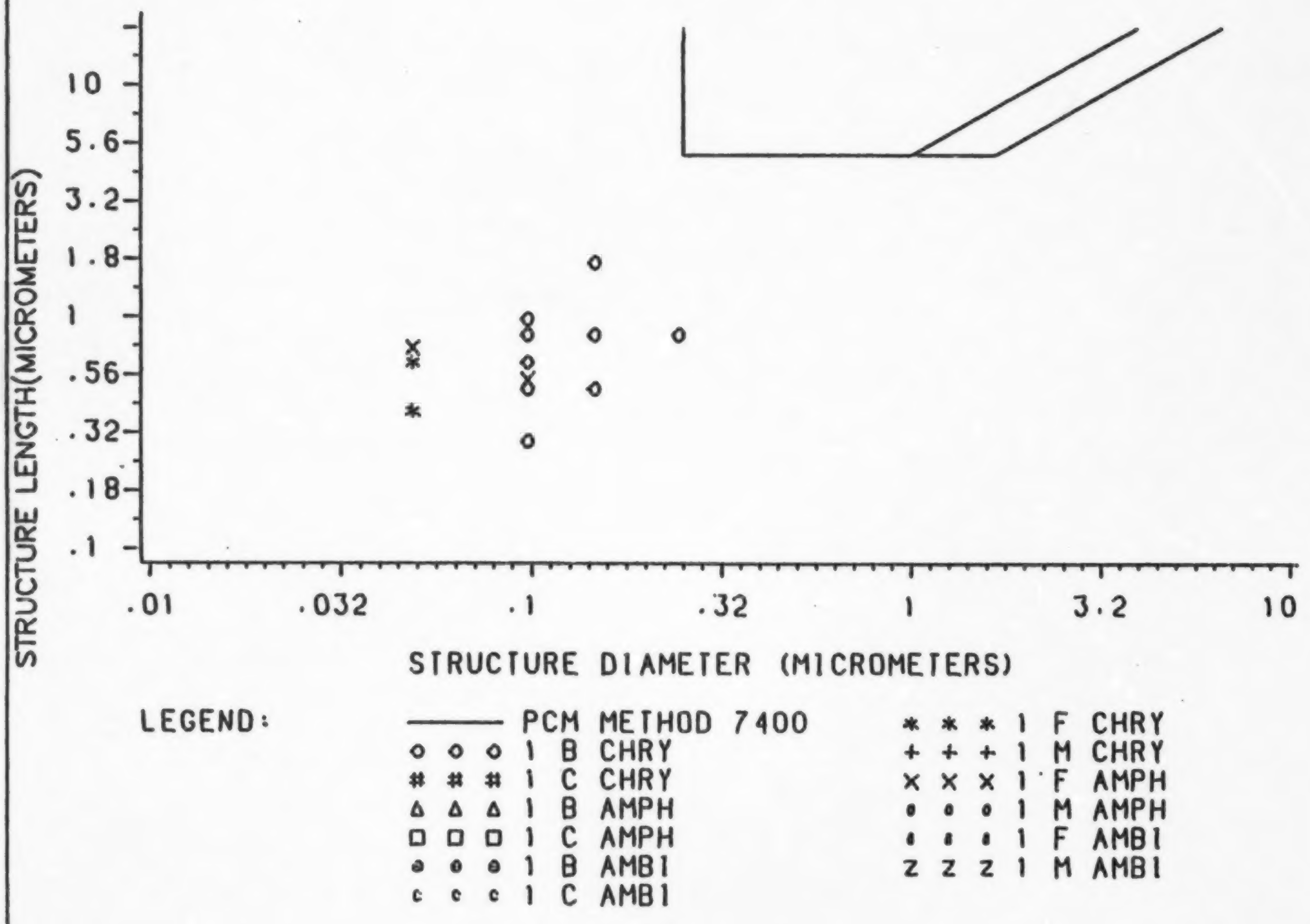
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS
FILTER=1163



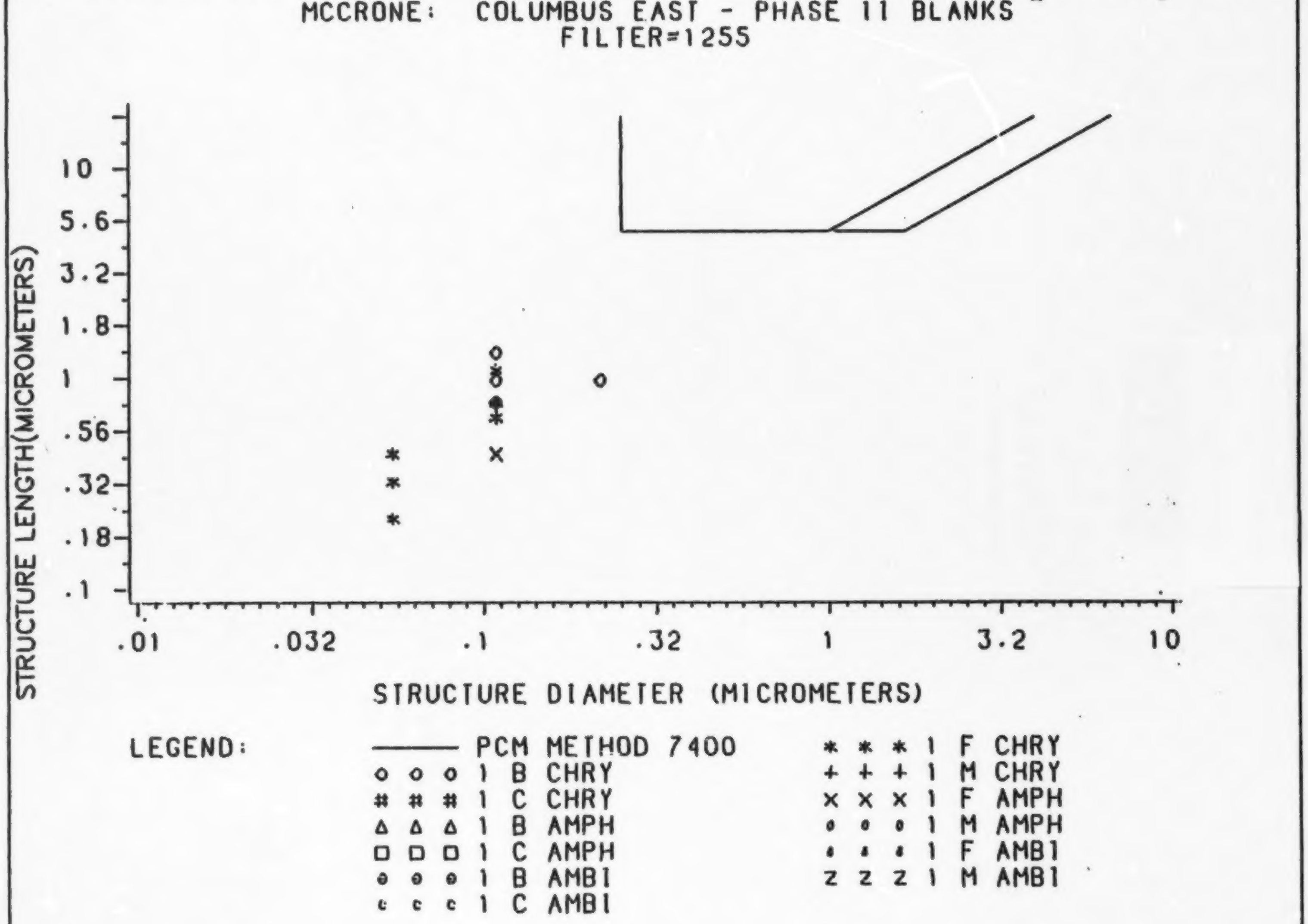
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS
FILTER=1173



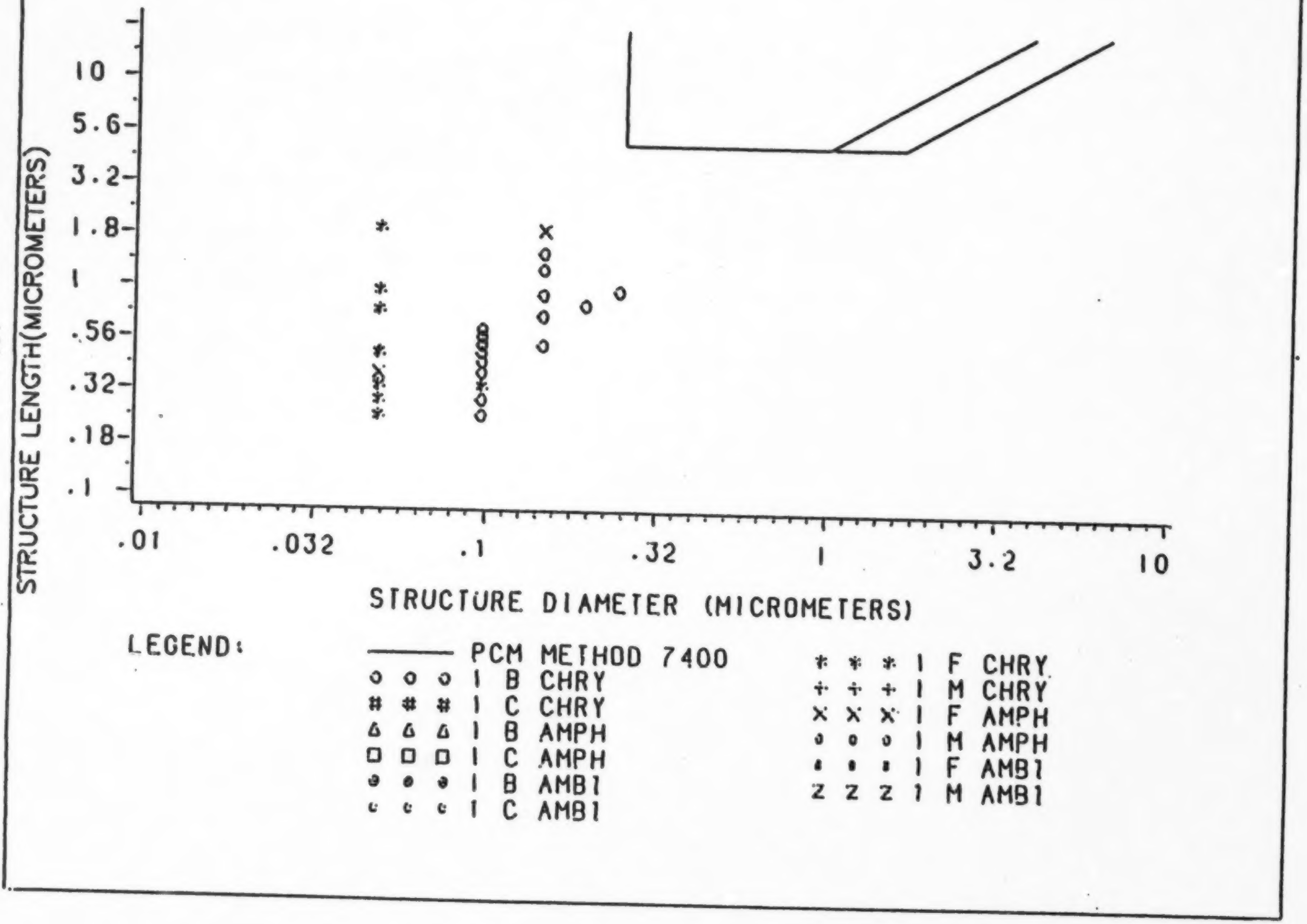
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE 11 BLANKS
FILTER=1255



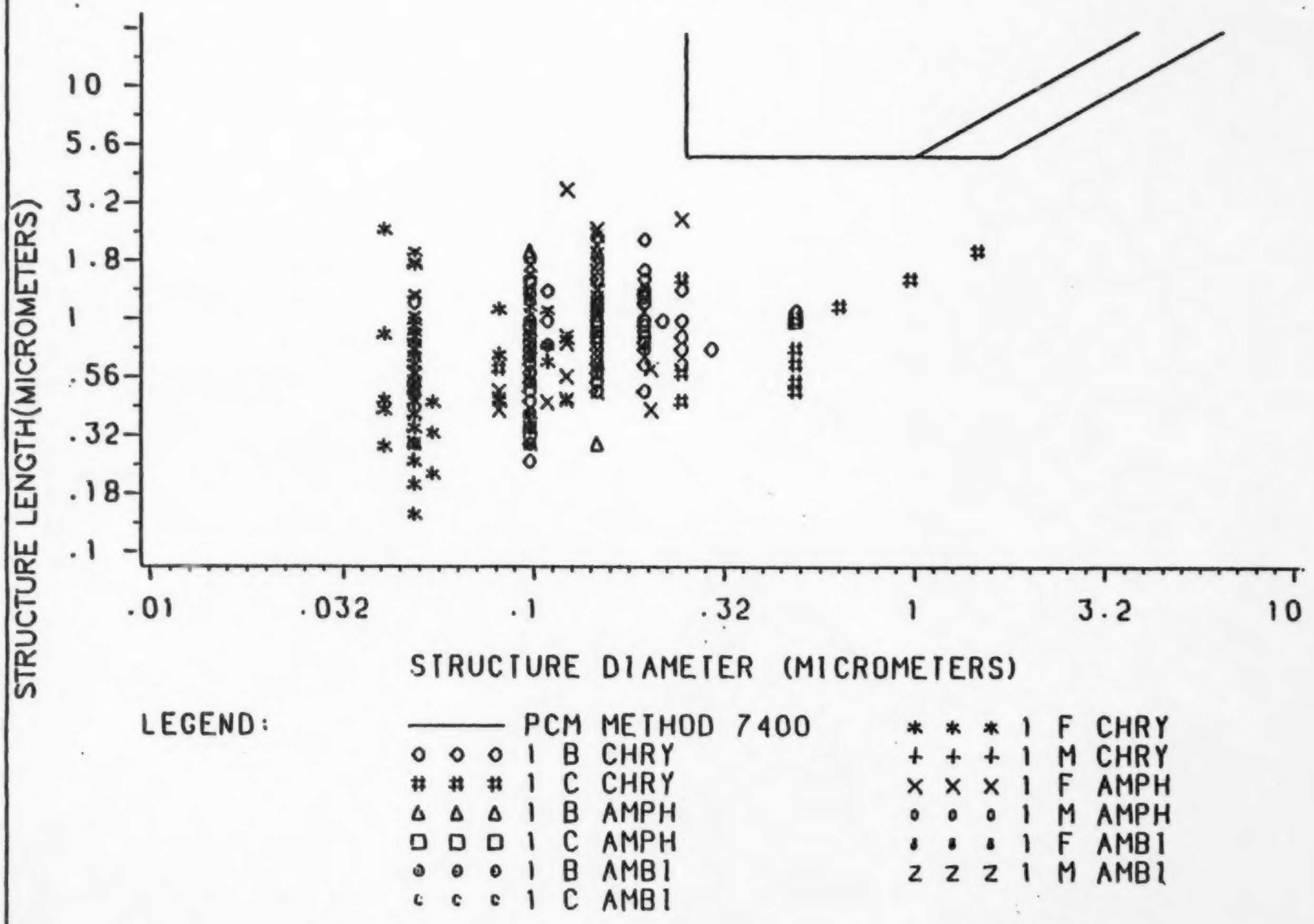
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS
FILTER=1255.1

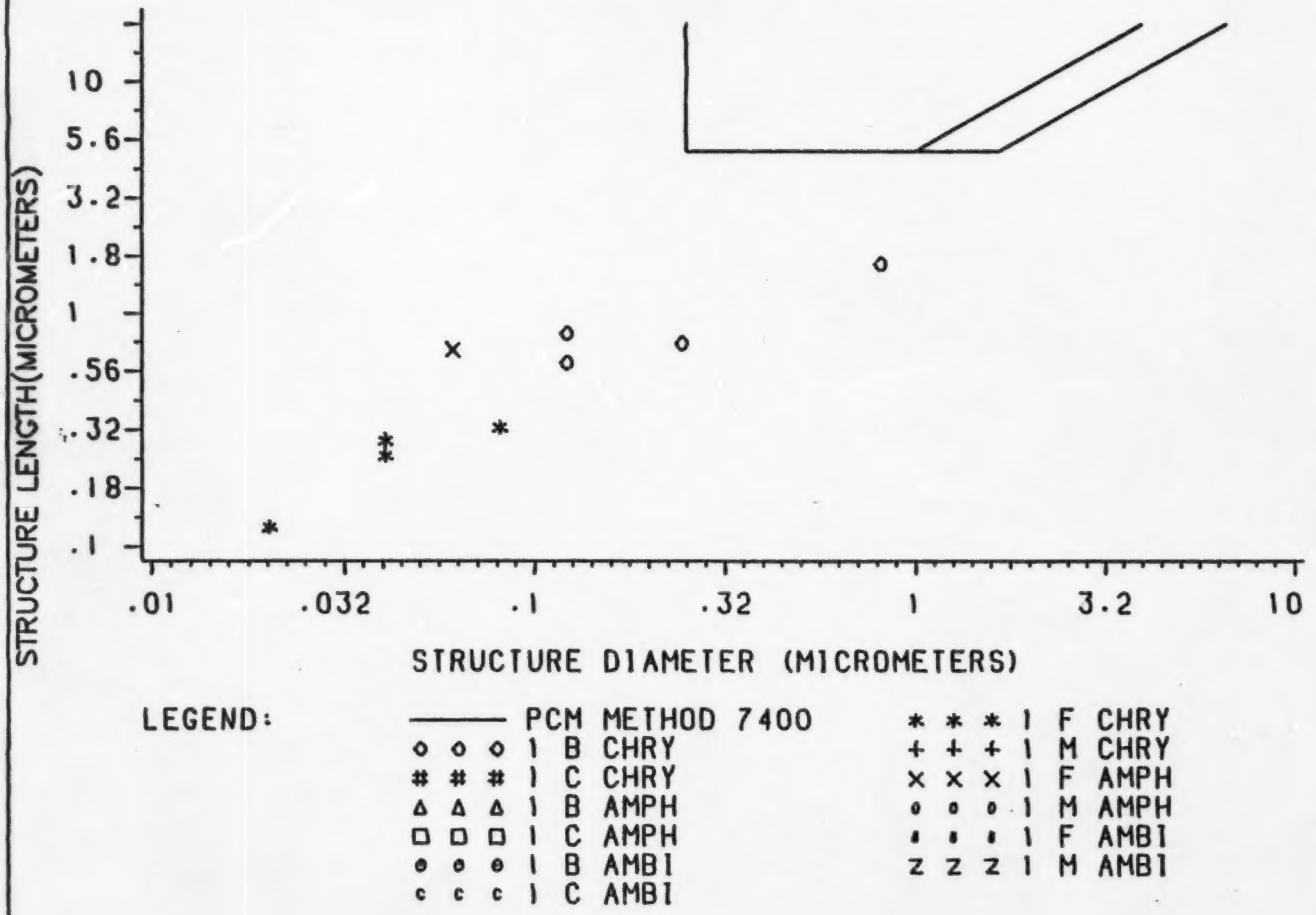


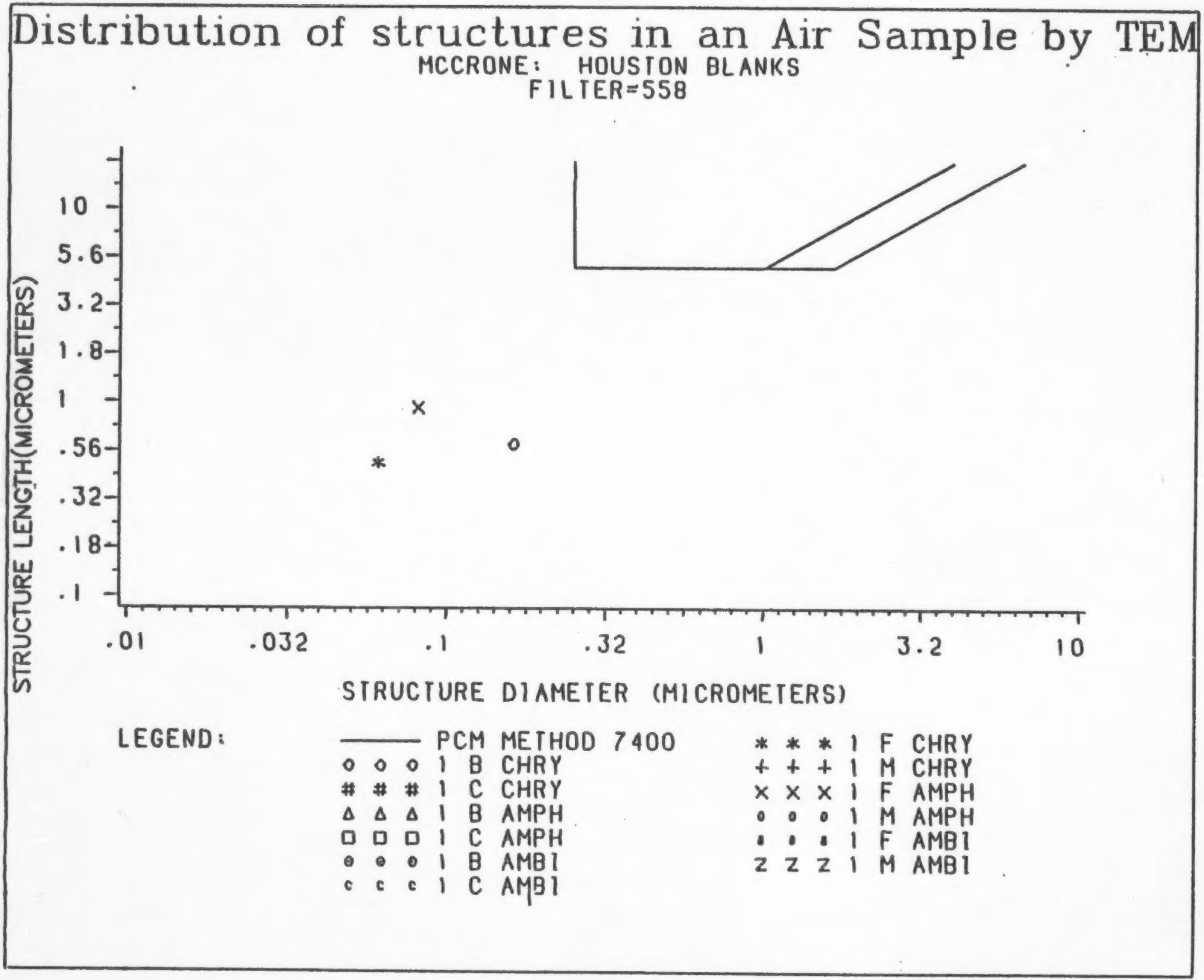
Distribution of Structures in an Air Sample By TEM

MCCRONE: COLUMBUS EAST - PHASE II BLANKS



Distribution of structures in an Air Sample by TEM
MCCRONE: HOUSTON BLANKS
FILTER=566

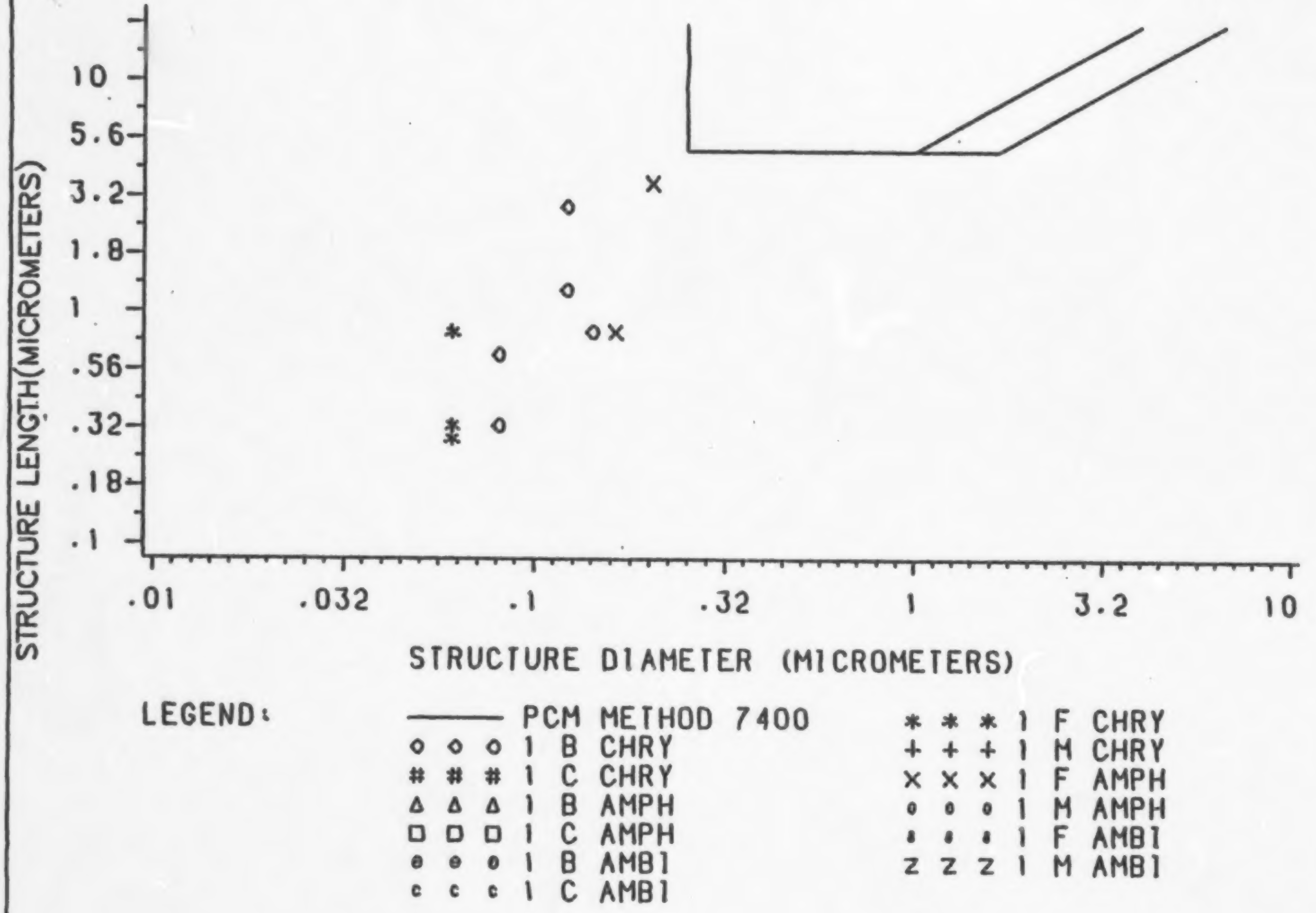



Distribution of structures in an Air Sample by TEM
MCCRONE: HOUSTON BLANKS
FILTER=558
STRUCTURE LENGTH(MICROMETERS)
10
5.6
3.2
1.8
1
.56
.32
.18
.1
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND:
——— PCM METHOD 7400
o o o 1 B CHRY
# 1 C CHRY
Δ Δ Δ 1 B AMPH
□ □ □ 1 C AMPH
⊖ ⊖ ⊖ 1 B AMBI
c c c 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
x x x 1 F AMPH
o o o 1 M AMPH
• • • 1 F AMBI
z z z 1 M AMBI

Distribution of structures in an Air Sample by TEM

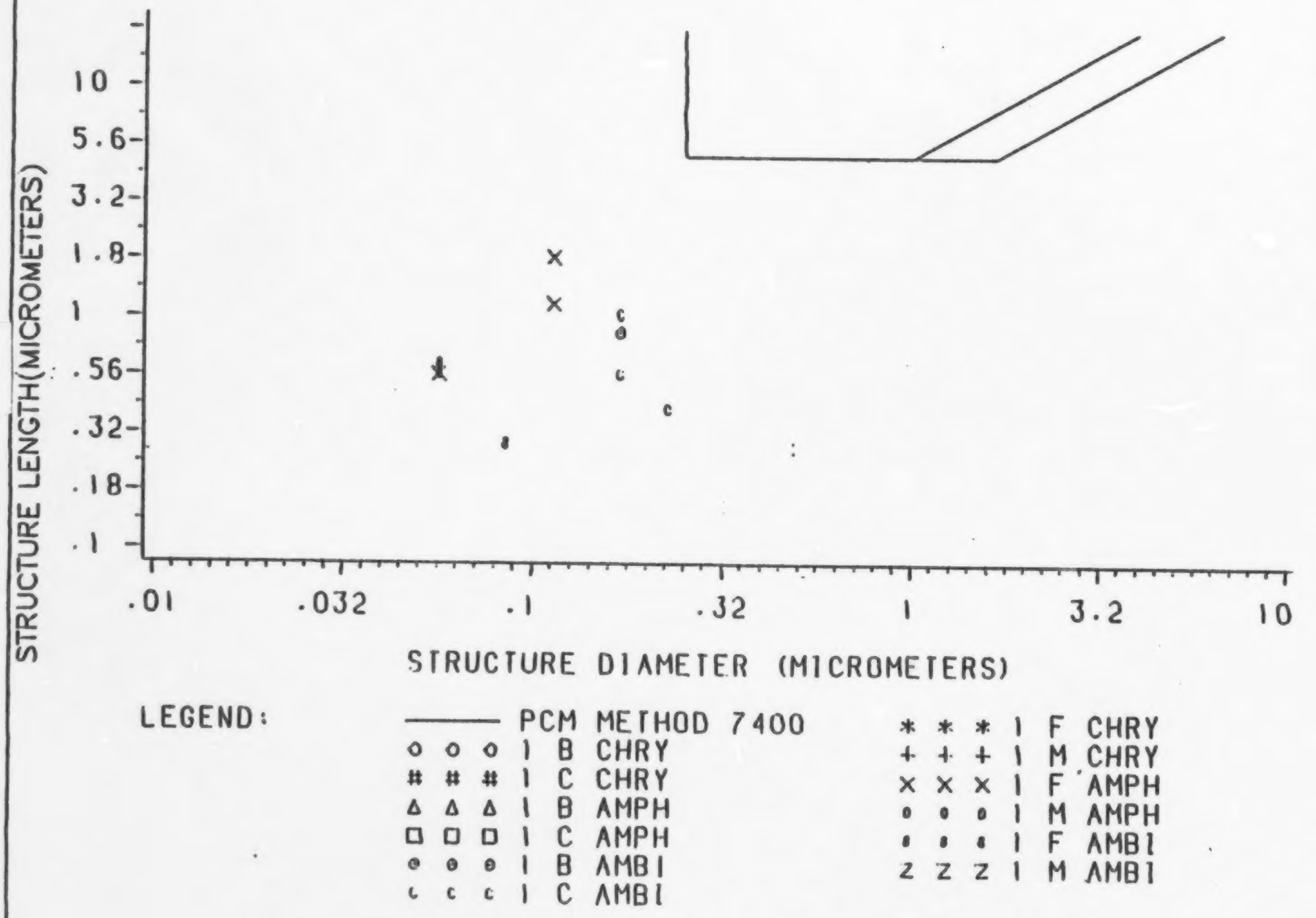
MCCRONE: HOUSTON BLANKS

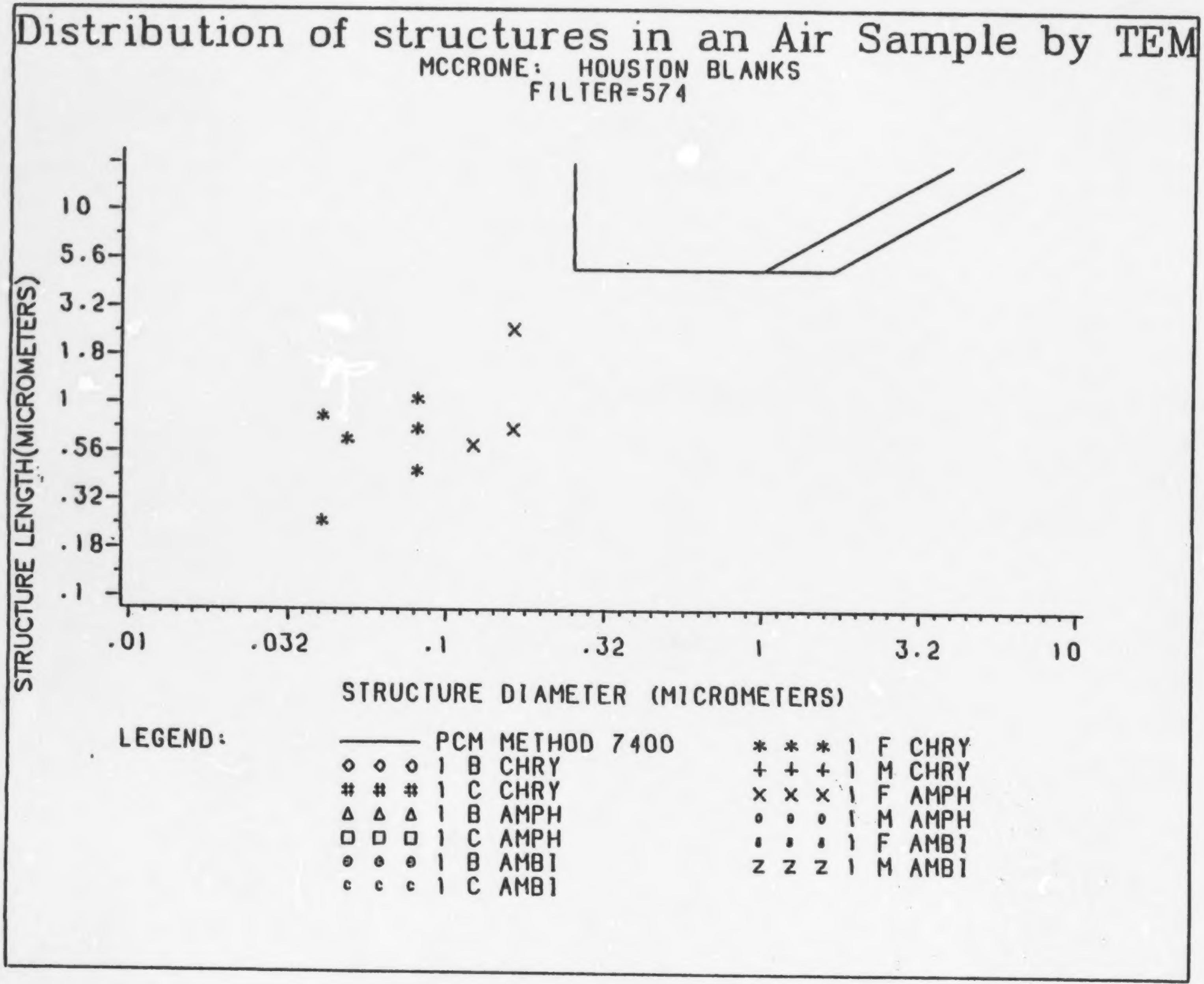
FILTER=500



Distribution of Structures in an Air Sample by TEM

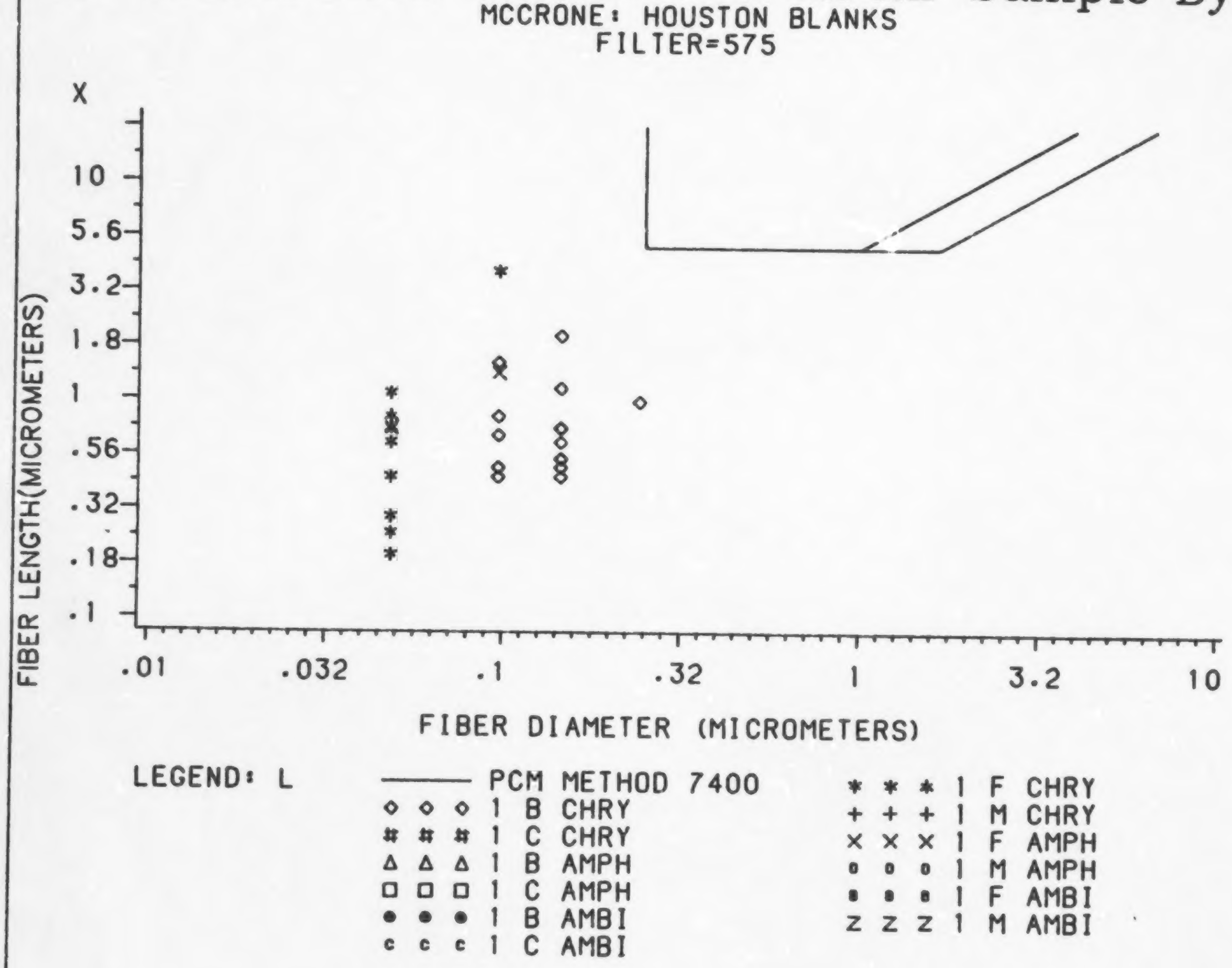
NIOSH: DUPLICATE ANALYSIS OF MCCRONE BLANKS
FILTER=550





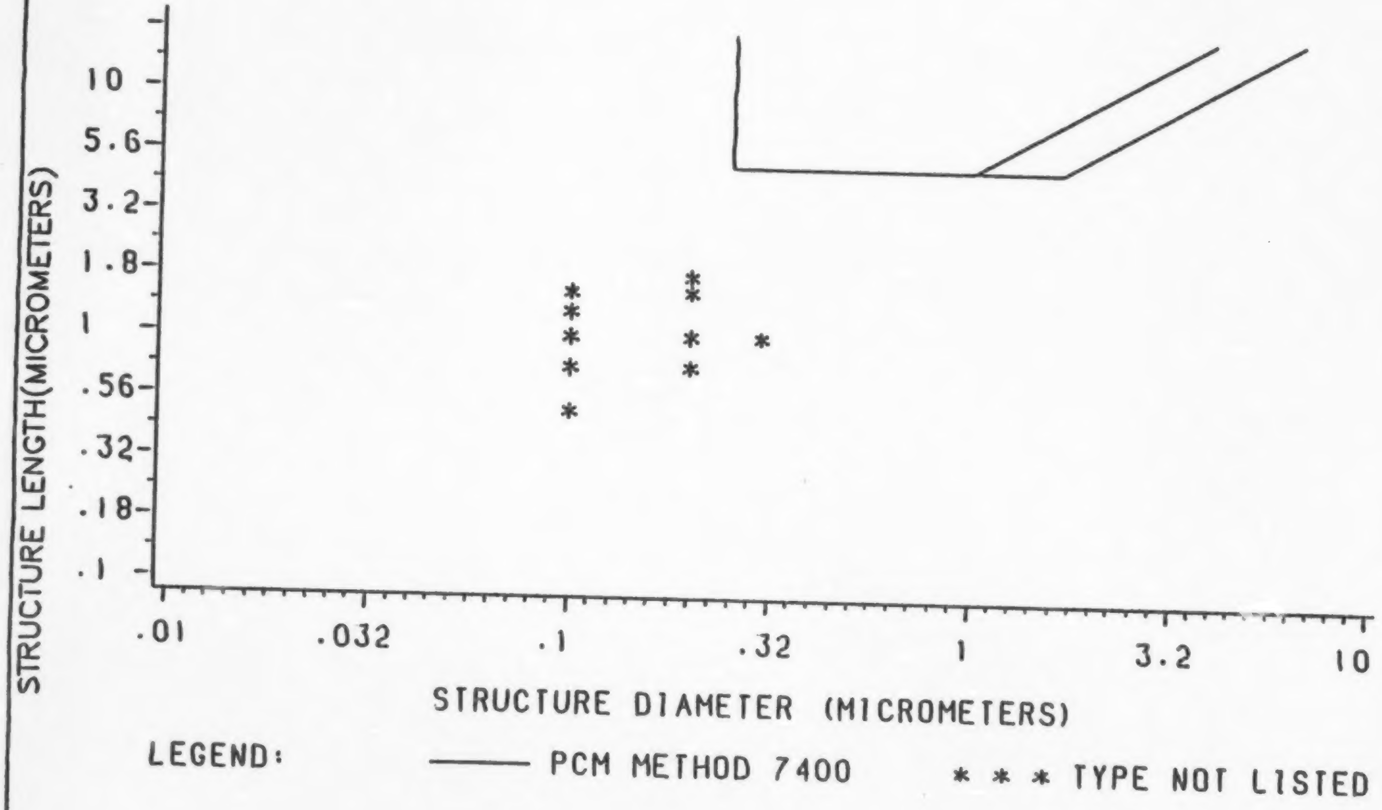
Distribution of structures in an Air Sample by TEM
MCCRONE: HOUSTON BLANKS
FILTER=574

Size Distribution of Fibers in an Air Sample By TEM



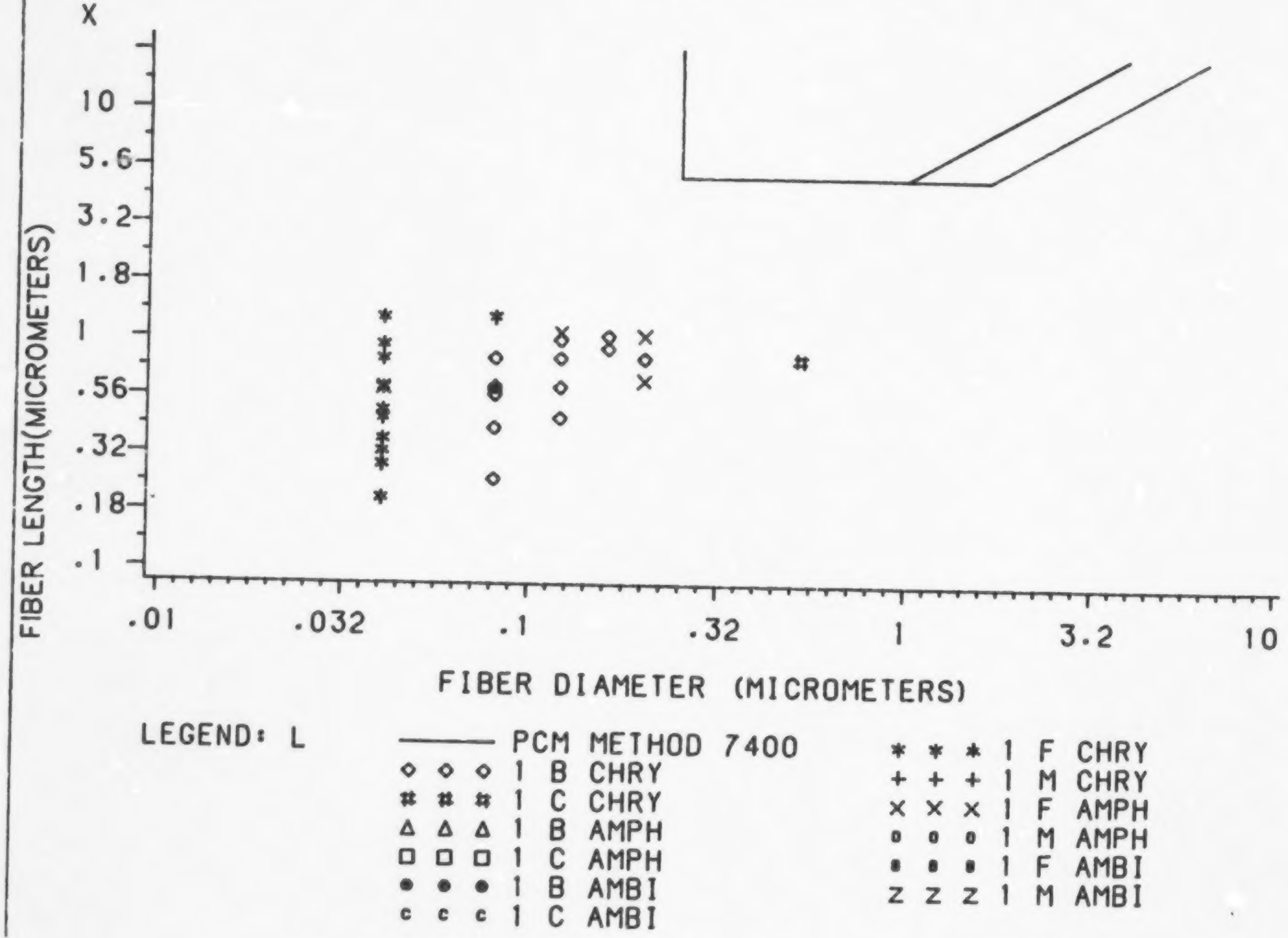
Distribution of Structures in an Air Sample by TEM

NIOSH: HOUSTON BLANKS. 116 GRID OPENINGS COUNTED
FILTER=575



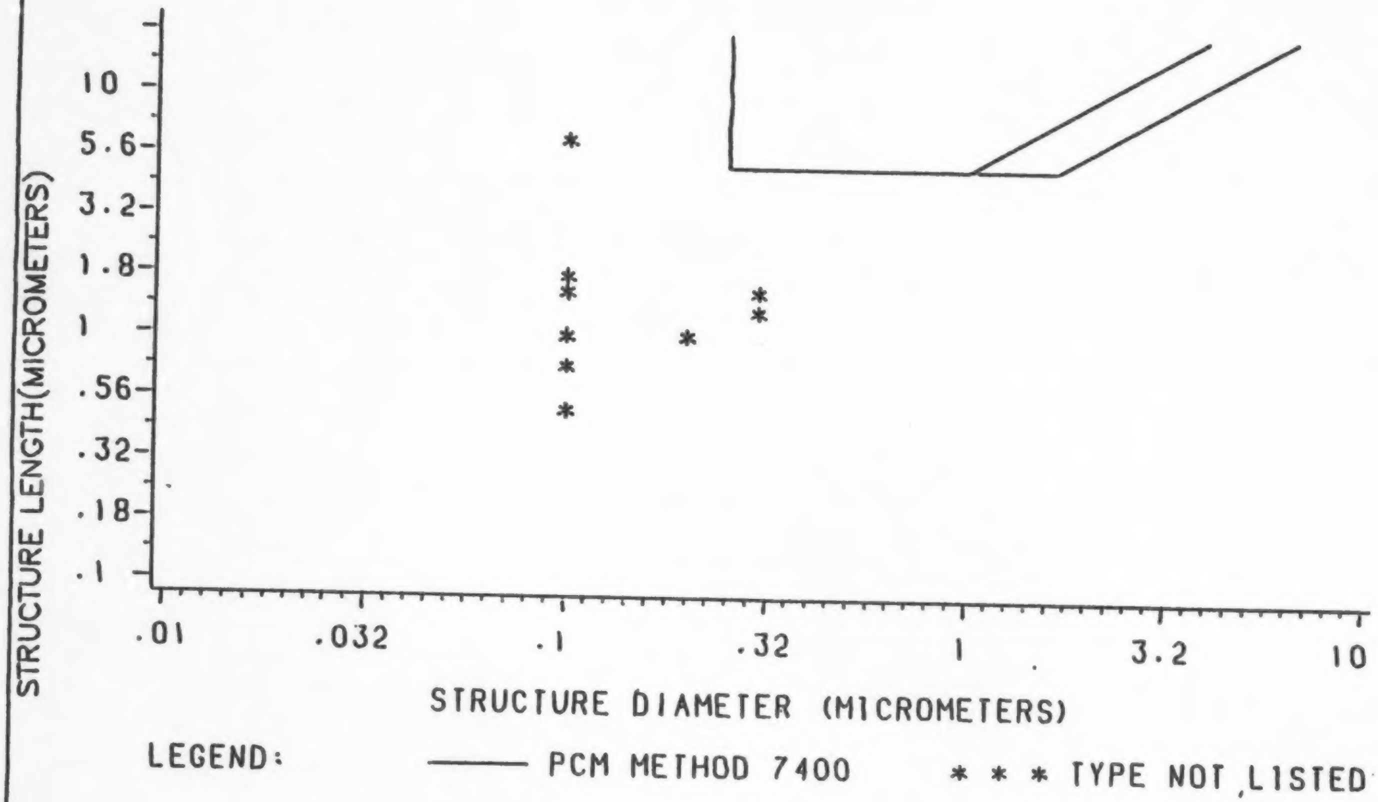
Size Distribution of Fibers in an Air Sample By TEM

MCCRONE: HOUSTON BLANKS
FILTER=576



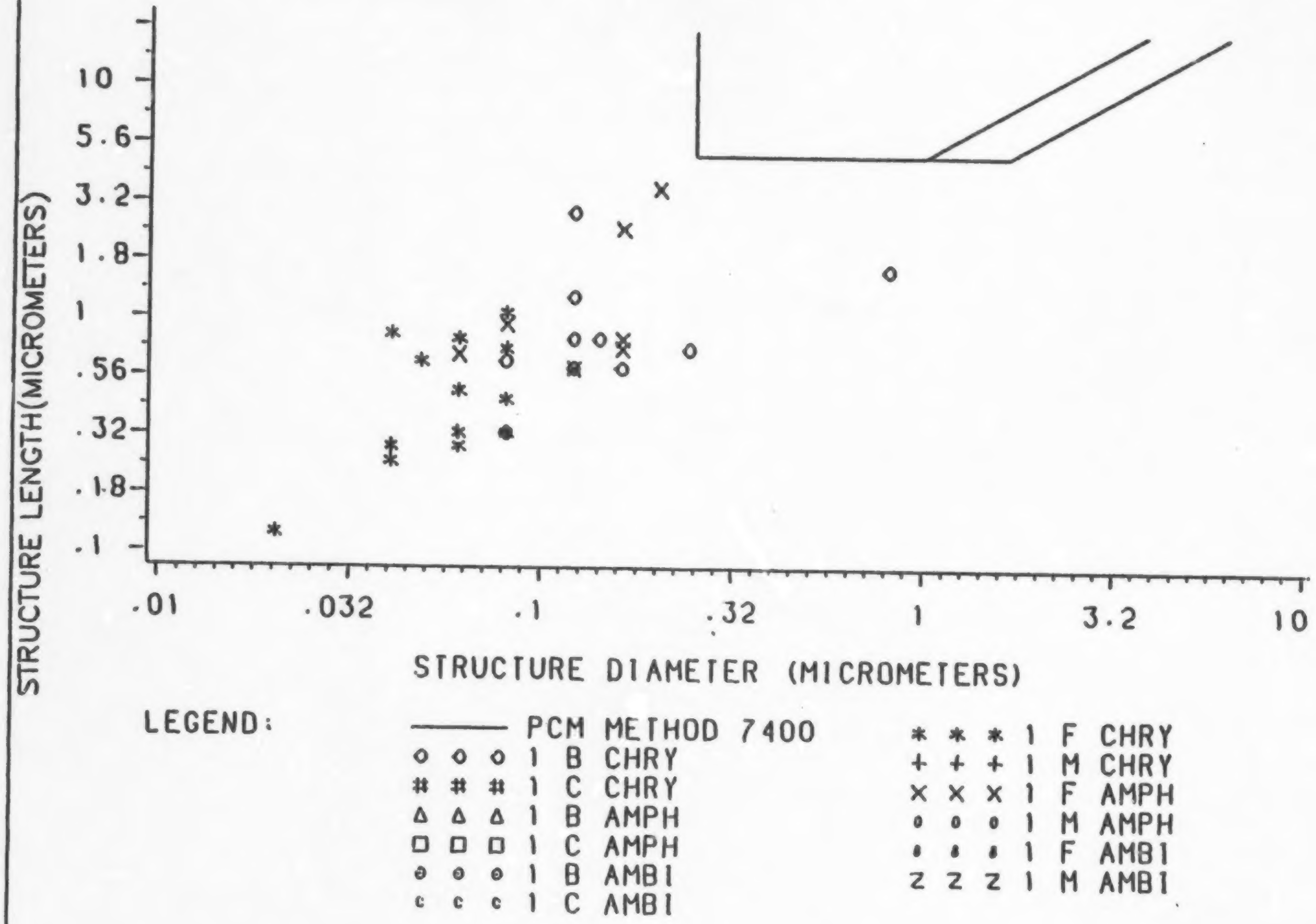
Distribution of Structures in an Air Sample by TEM

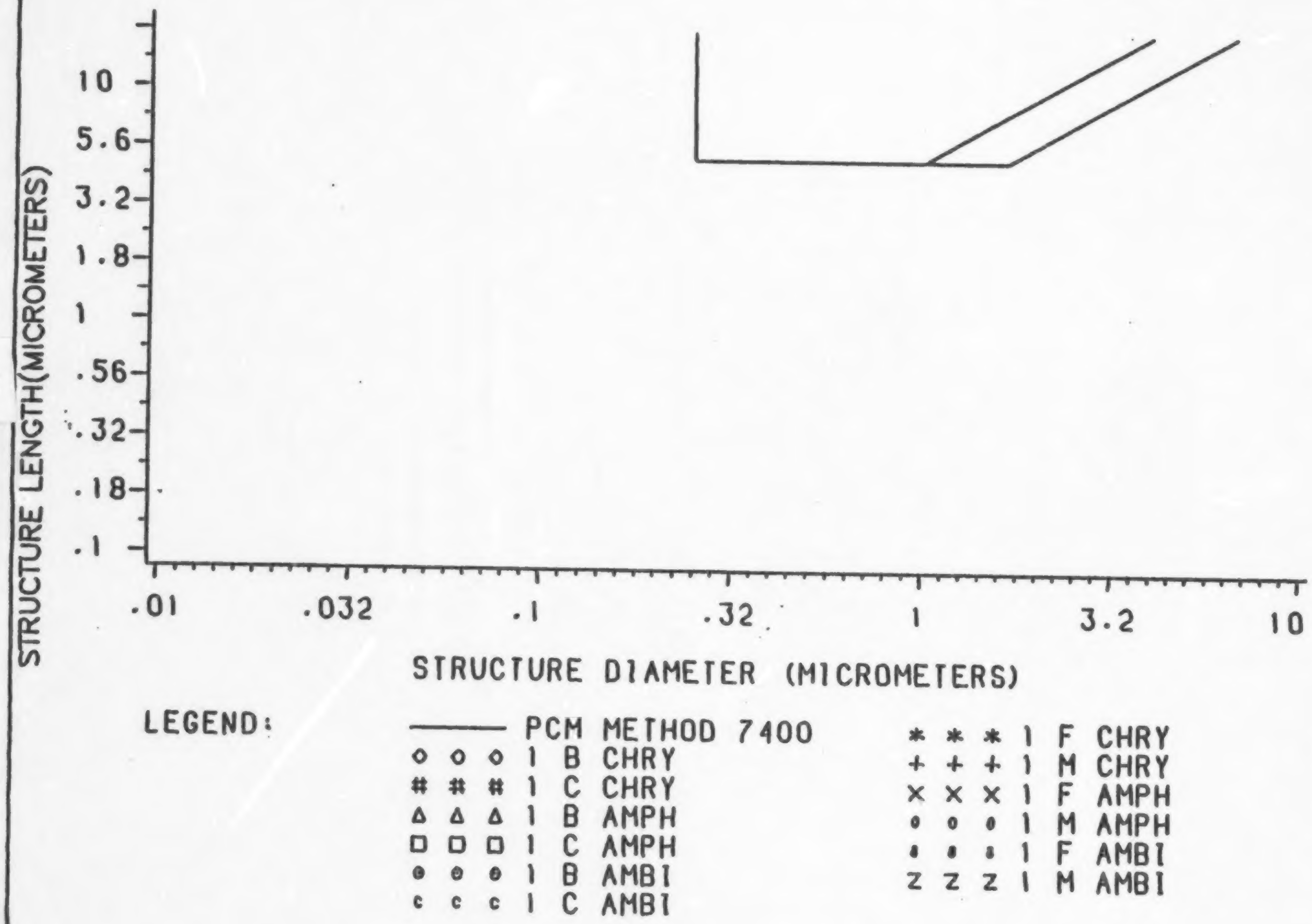
NIOSH: HOUSTON BLANKS. 116 GRID OPENINGS COUNTED
FILTER=576



Distribution of structures in an Air Sample by TEM

MCCRONE: HOUSTON BLANKS - PREABATEMENT



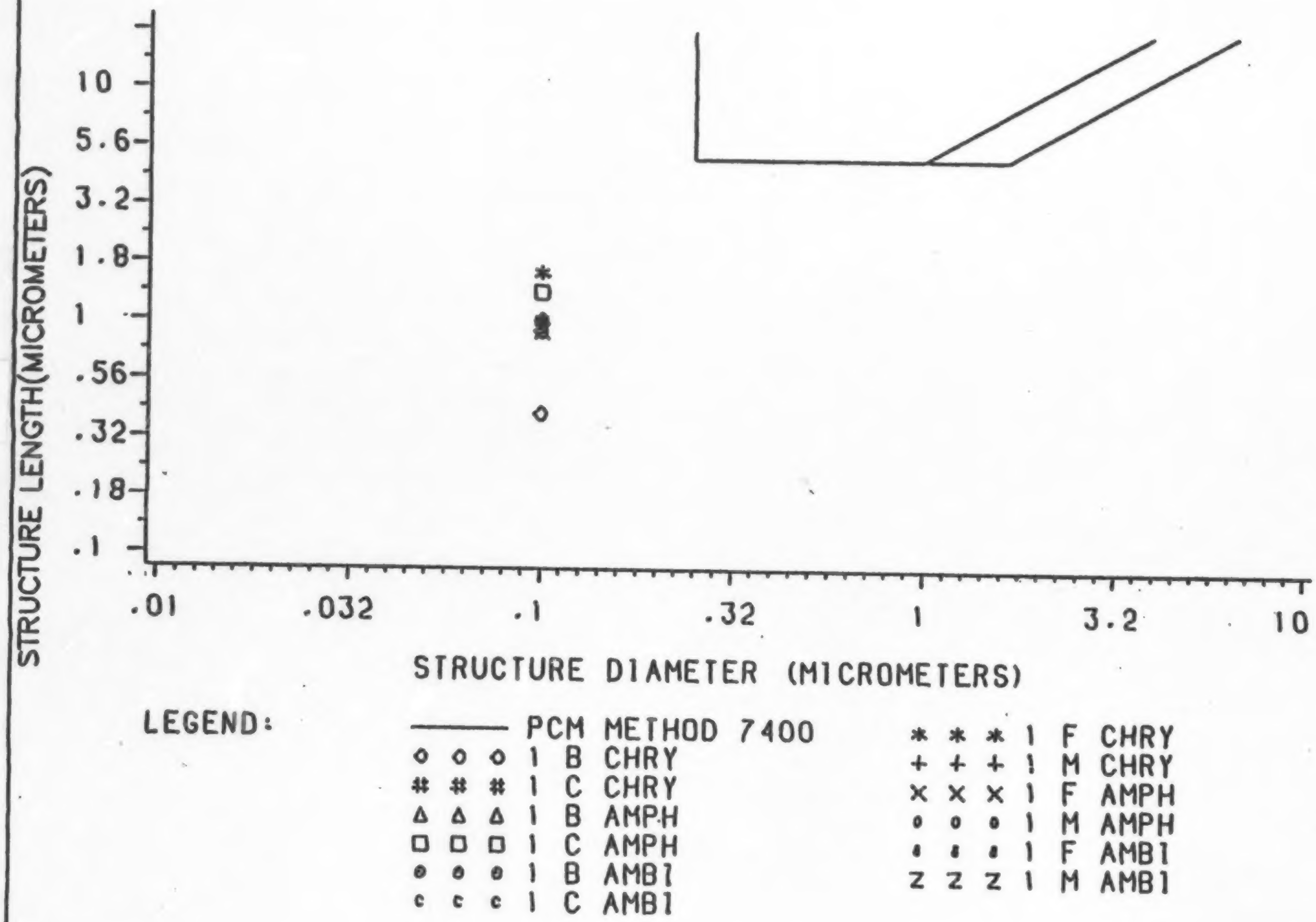


Distribution of Structures in an Air Sample By TEM

ETC: COLUMBUS EAST BLANKS
FILTER=1175

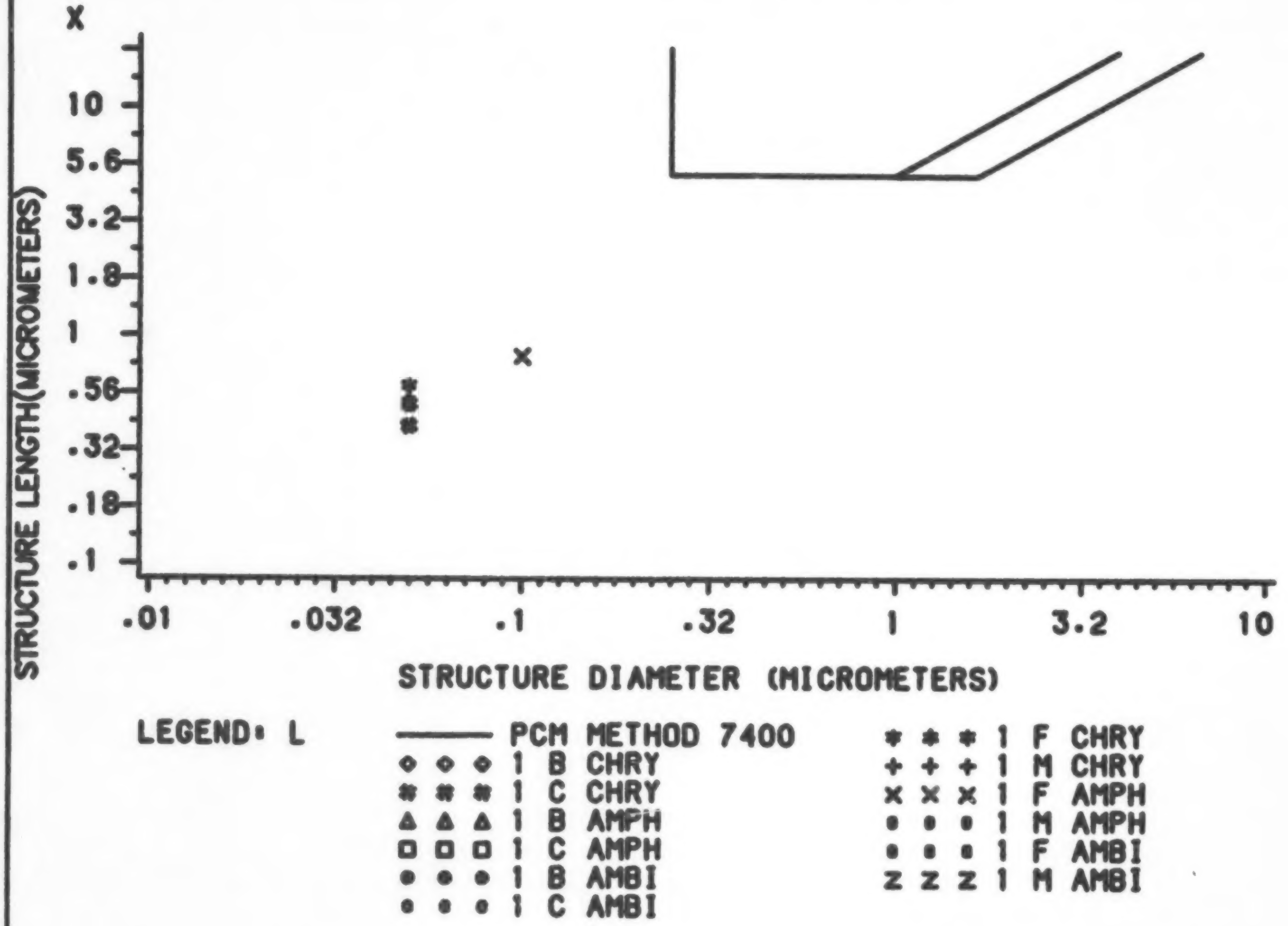
Distribution of Structures in an Air Sample By TEM

ETC: COLUMBUS EAST BLANKS
FILTER=1176



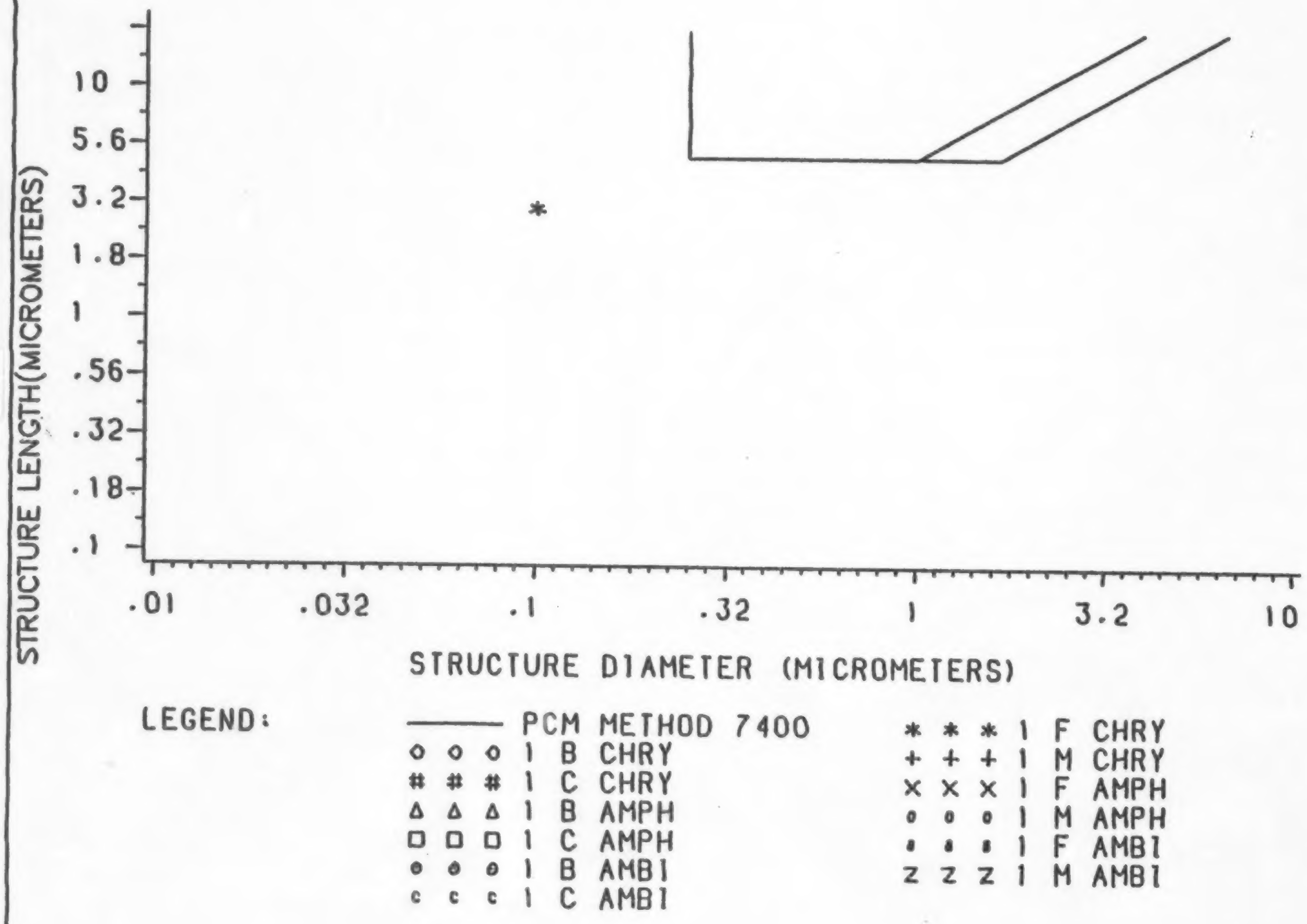
Distribution of Structures in an Air Sample by TEM

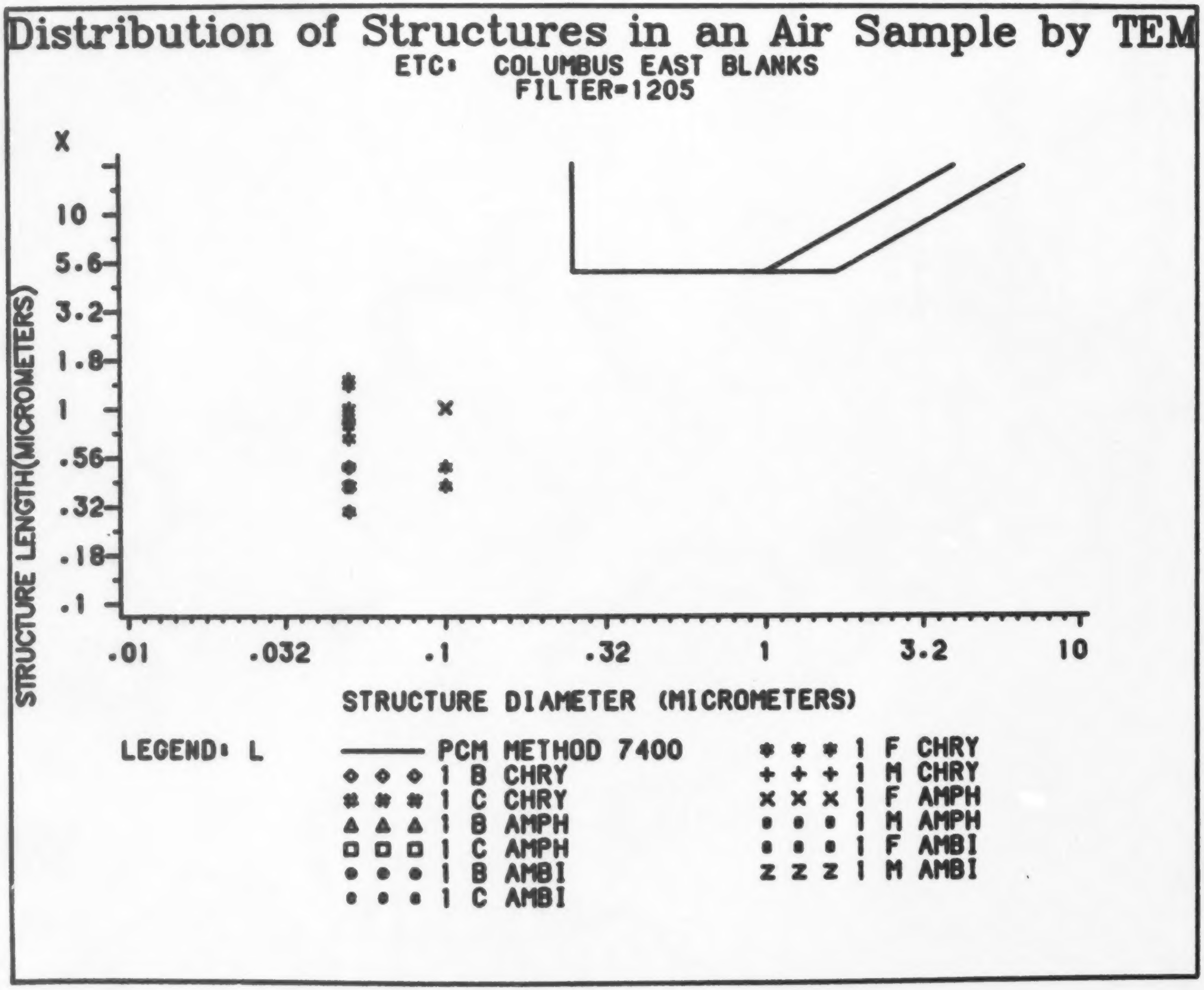
ETC: COLUMBUS EAST BLANKS
FILTER=1177

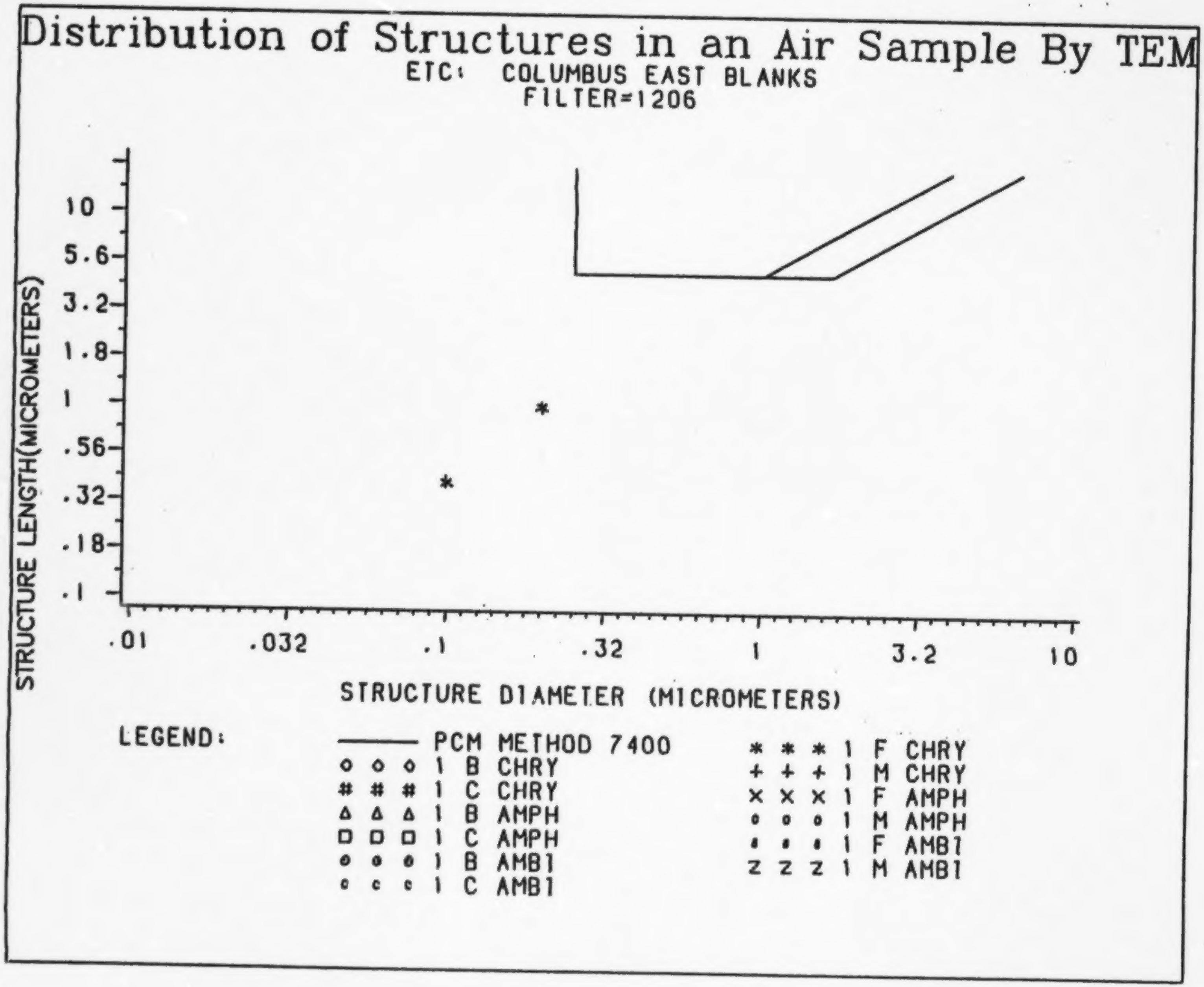


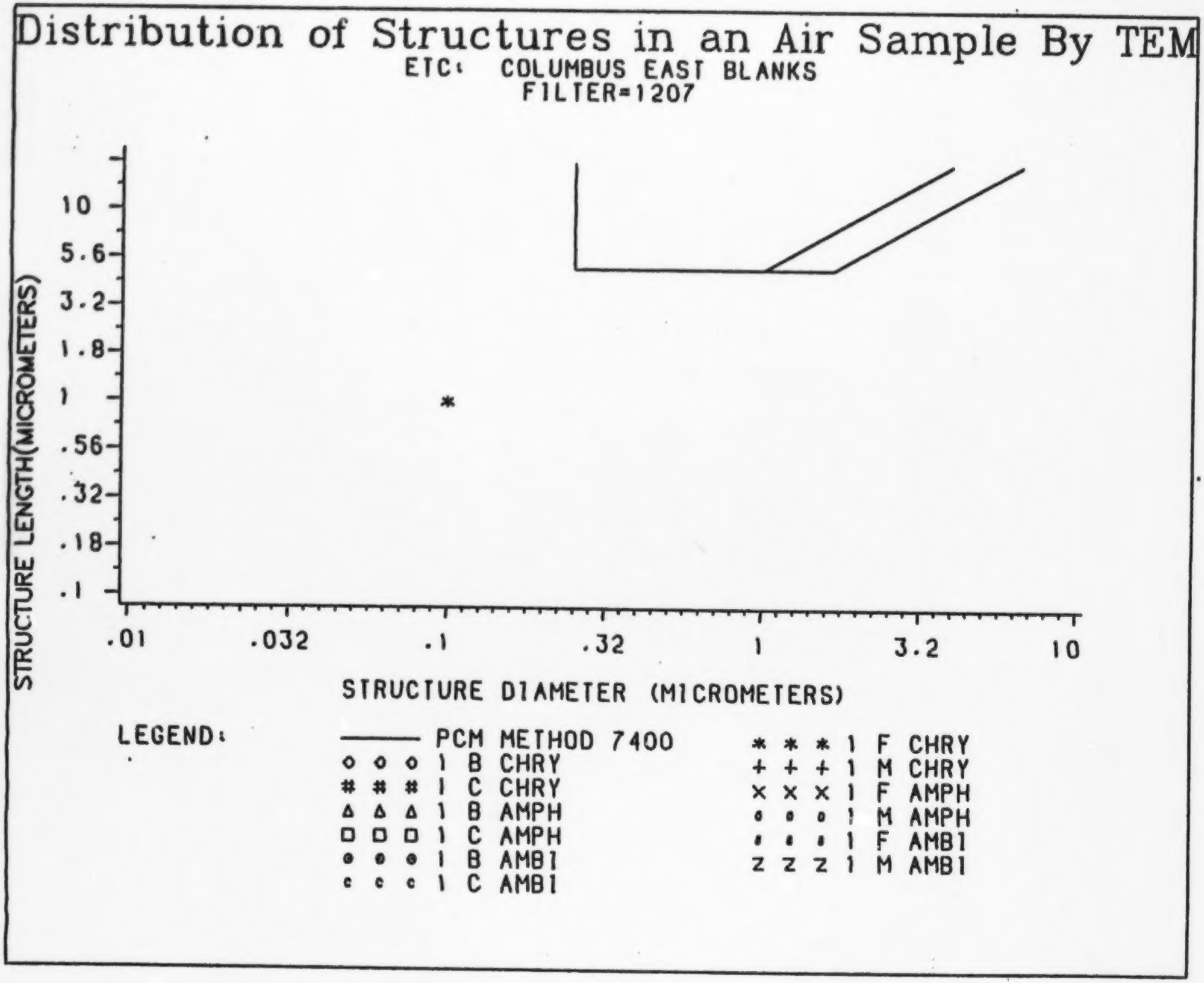
Distribution of Structures in an Air Sample By TEM

ETC: COLUMBUS EAST BLANKS
FILTER=1204




Distribution of Structures in an Air Sample by TEM
ETC: COLUMBUS EAST BLANKS
FILTER=1205
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
STRUCTURE LENGTH(MICROMETERS)
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
PCM METHOD 7400
◇ ◇ ◇ 1 B CHRY
# 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
• • • 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
▪ ▪ ▪ 1 M AMPH
▪ ▪ ▪ 1 F AMBI
Z Z Z 1 M AMBI


Distribution of Structures in an Air Sample By TEM
ETC: COLUMBUS EAST BLANKS
FILTER=1206
STRUCTURE LENGTH(MICROMETERS)
10
5.6
3.2
1.8
1
.56
.32
.18
.1
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND:
——— PCM METHOD 7400
○ ○ ○ 1 B CHRY
# 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMB1
● ● ● 1 C AMB1
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
● ● ● 1 M AMPH
● ● ● 1 F AMB1
Z Z Z 1 M AMB1



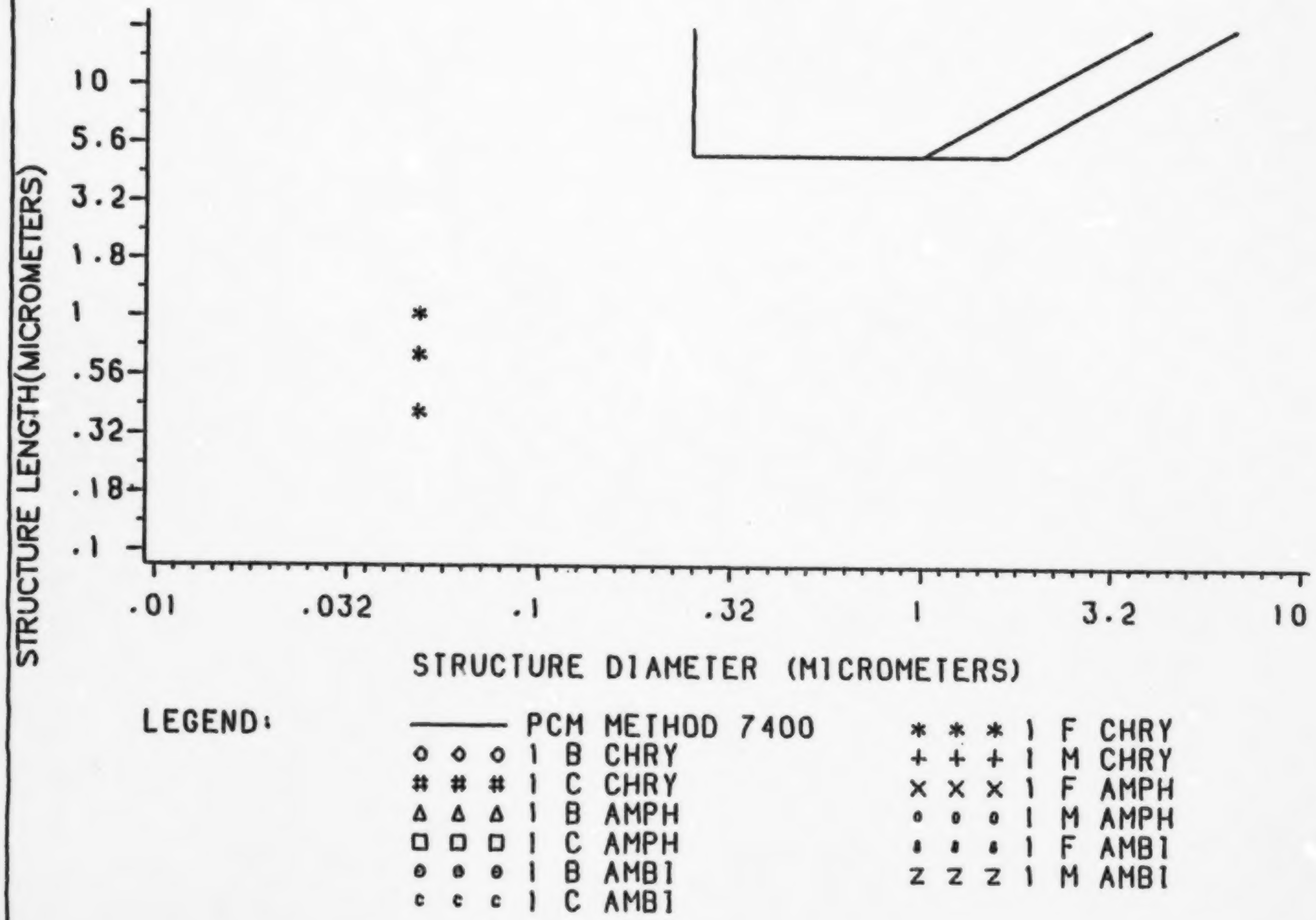
Distribution of Structures in an Air Sample By TEM

ETC: COLUMBUS EAST BLANKS

FILTER=1207

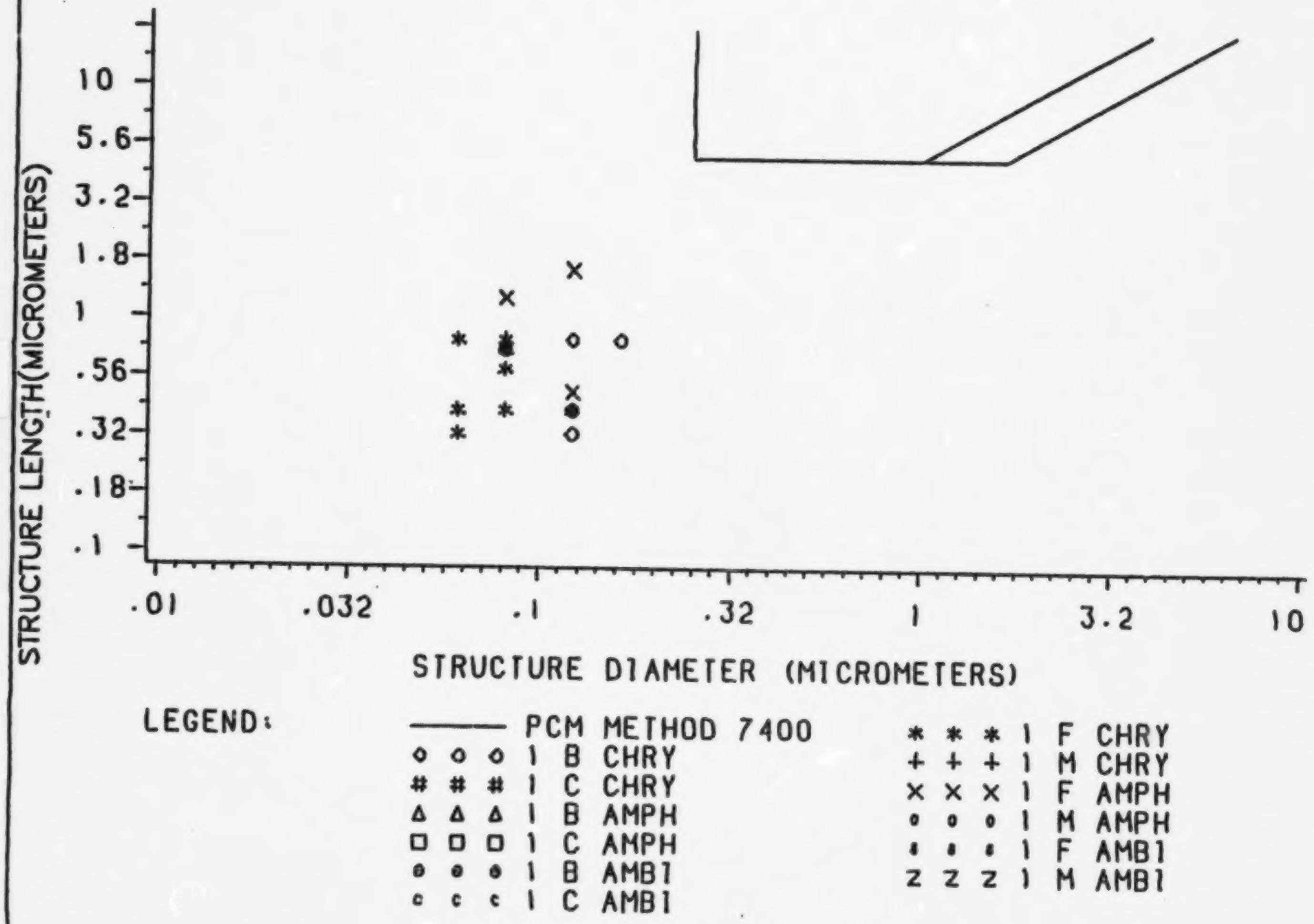
Distribution of structures in an Air Sample by TEM

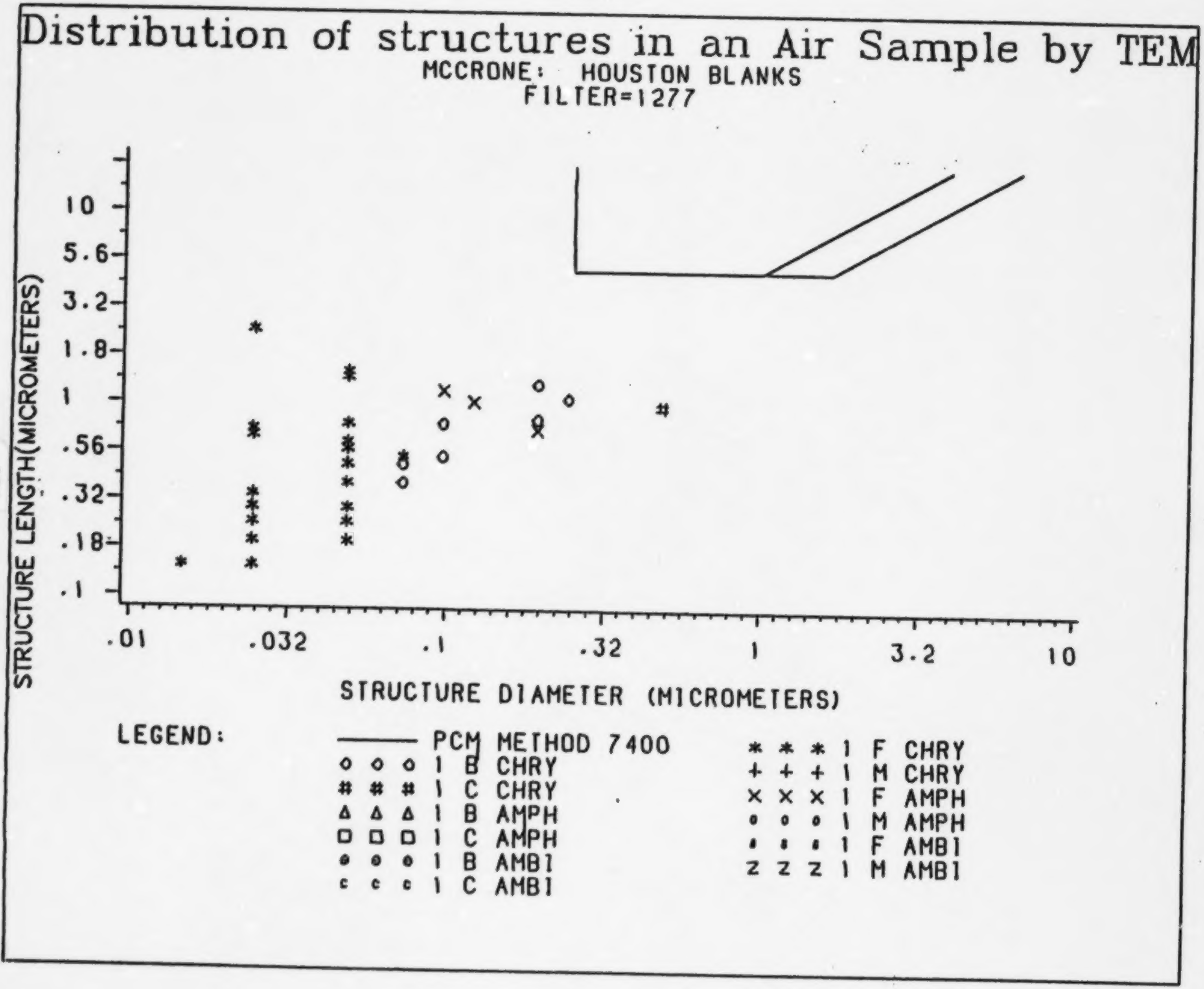
MCCRONE: HOUSTON BLANKS
FILTER=1263

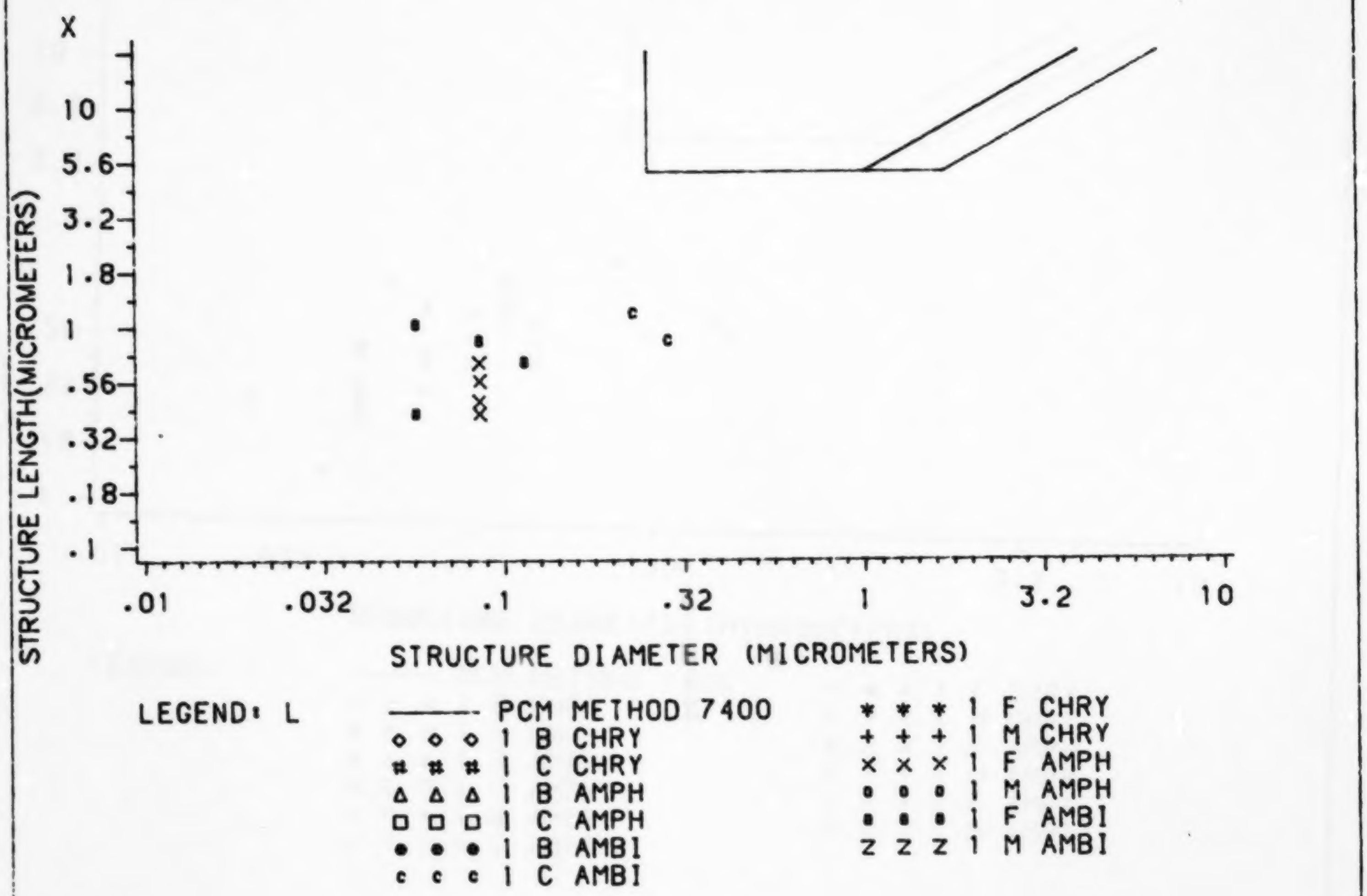


Distribution of structures in an Air Sample by TEM

MCCRONE: HOUSTON BLANKS
FILTER=1271



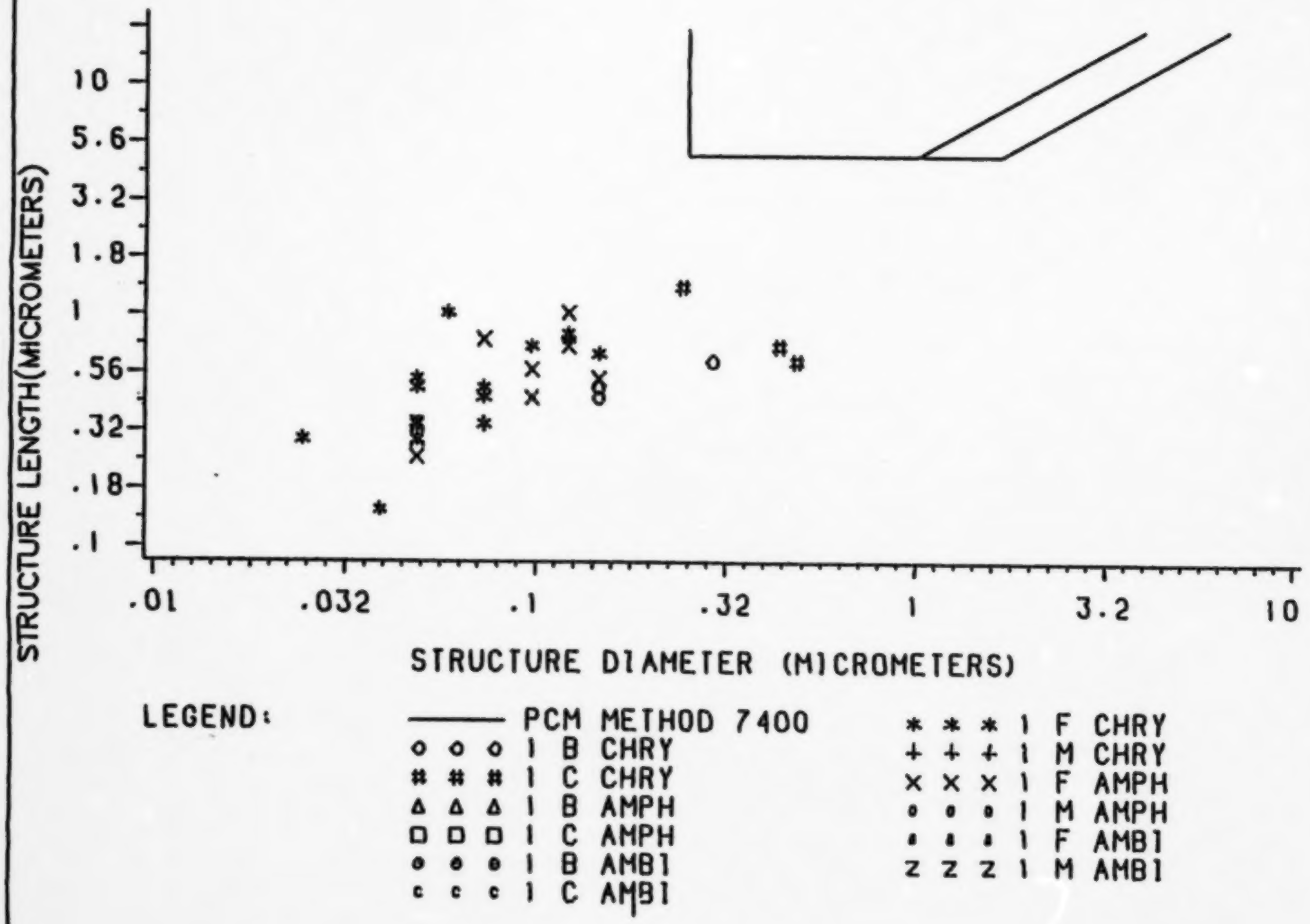

Distribution of structures in an Air Sample by TEM
MCCRONE: HOUSTON BLANKS
FILTER=1277



Distribution of Structures in an Air Sample by TEM
DUPLICATE ANALYSIS OF MCCRONE BLANKS
FILTER=1277

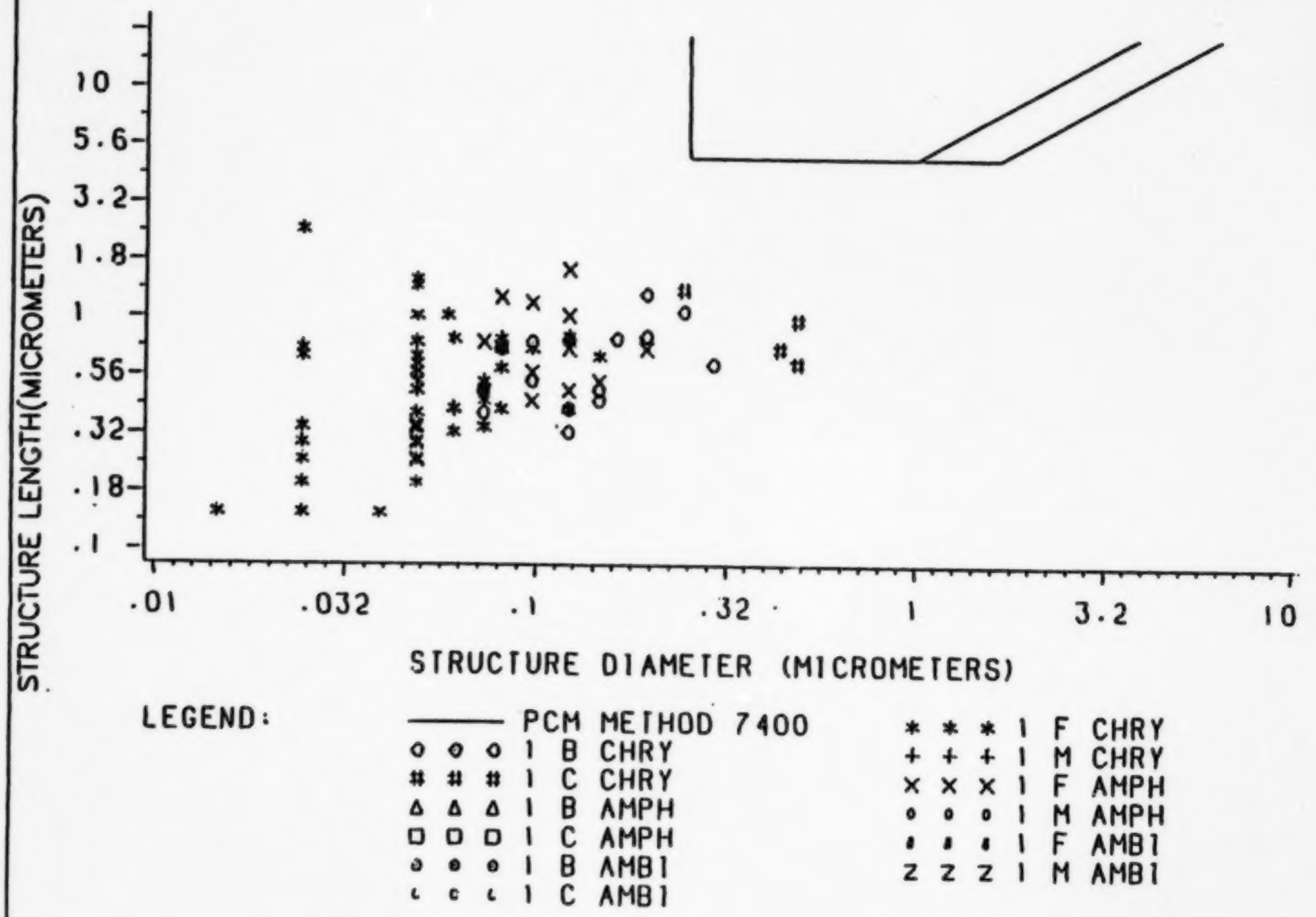
Distribution of structures in an Air Sample by TEM

MCCRONE: HOUSTON BLANKS
FILTER=1287



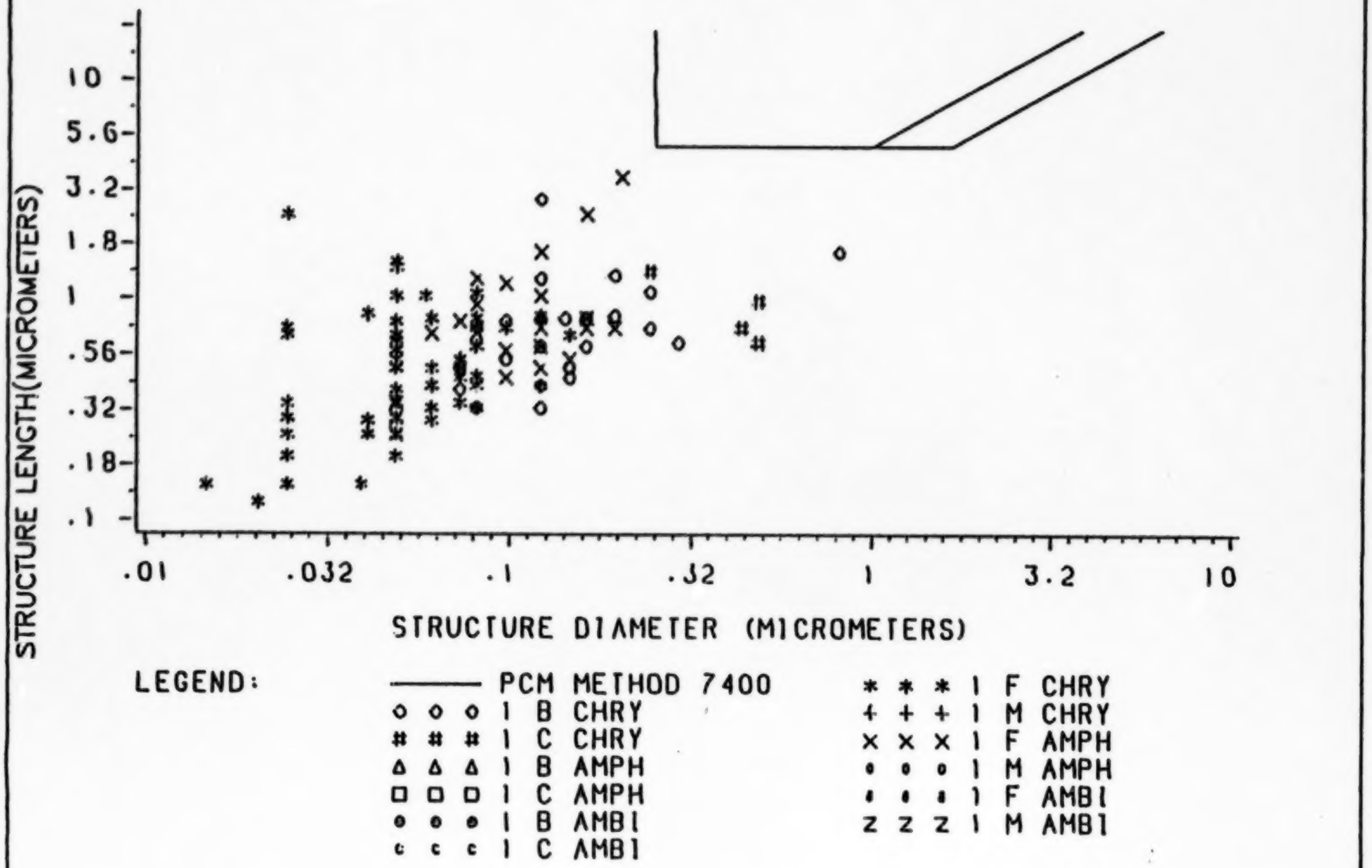
Distribution of structures in an Air Sample by TEM

MCCRONE: HOUSTON BLANKS - POSTABATEMENT



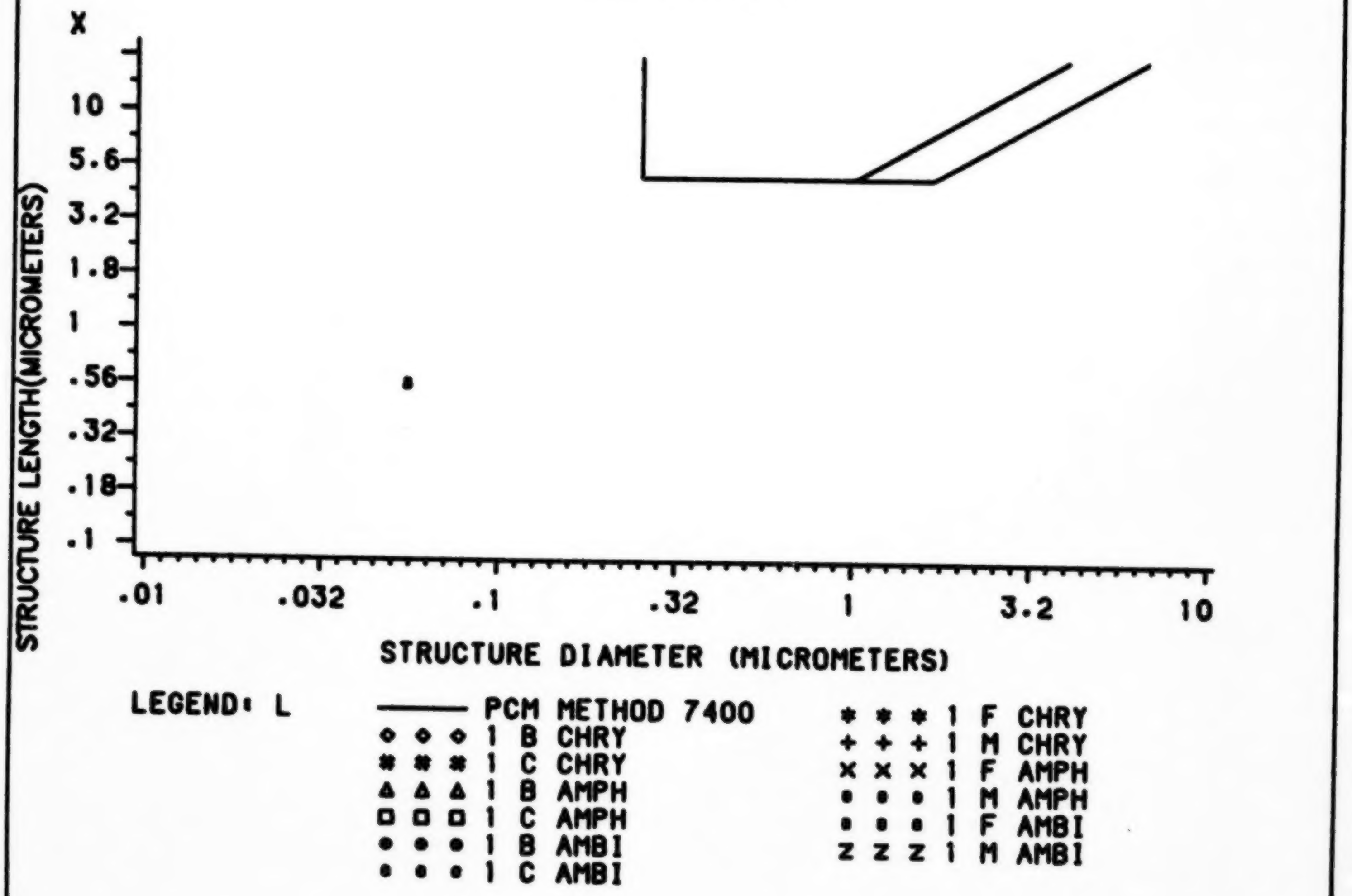
Distribution of structures in an Air Sample by TEM

MCCRONE: HOUSTON BLANKS



Distribution of Structures in an Air Sample by TEM

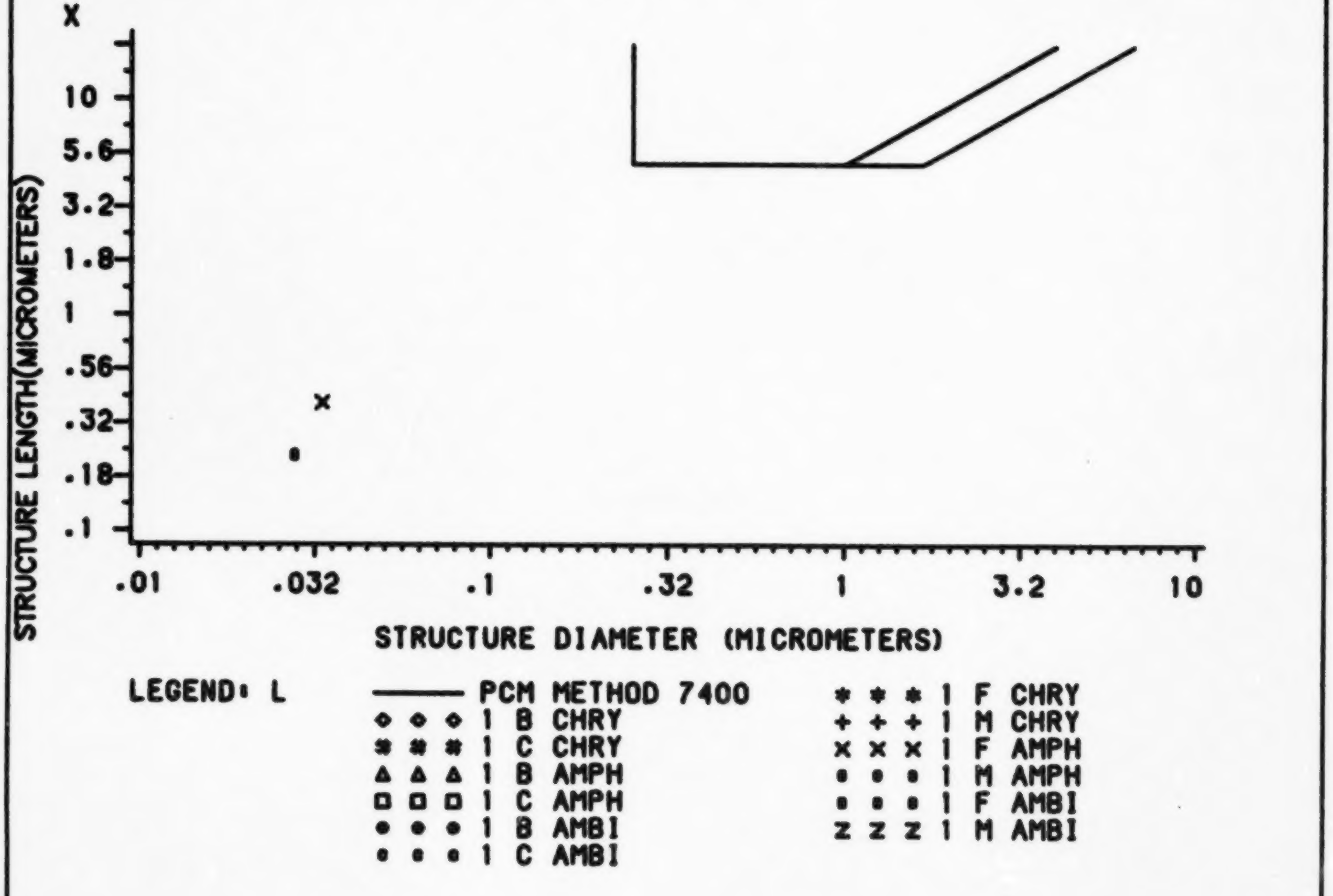
NIOSH: POSTABATEMENT COLUMBUS EAST PHASE II BLANKS
FILTER=1350



Distribution of Structures in an Air Sample by TEM

NIOSH: POSTABATEMENT COLUMBUS EAST PHASE II BLANKS

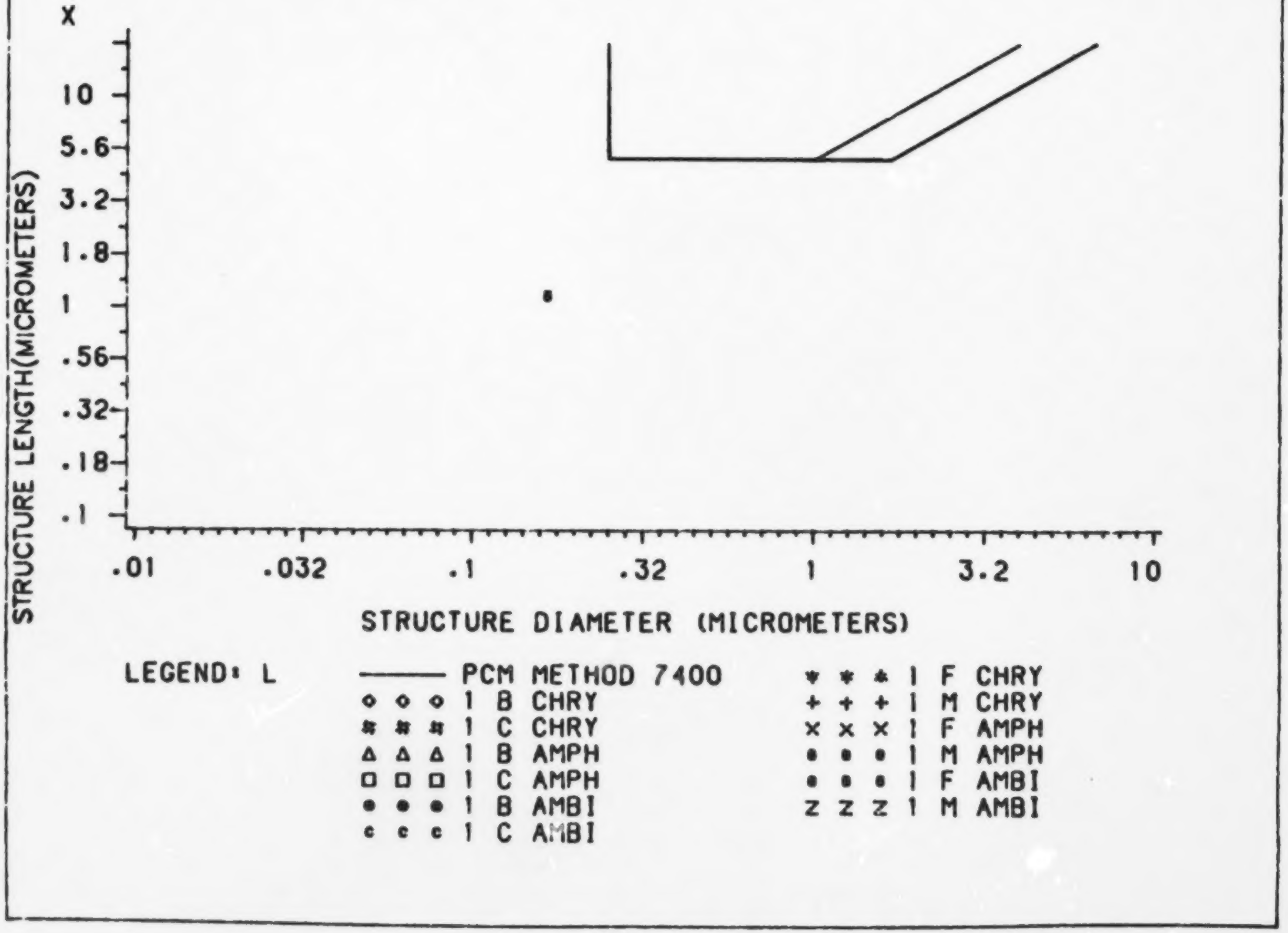
FILTER=1351



Distribution of Structures in an Air Sample by TEM

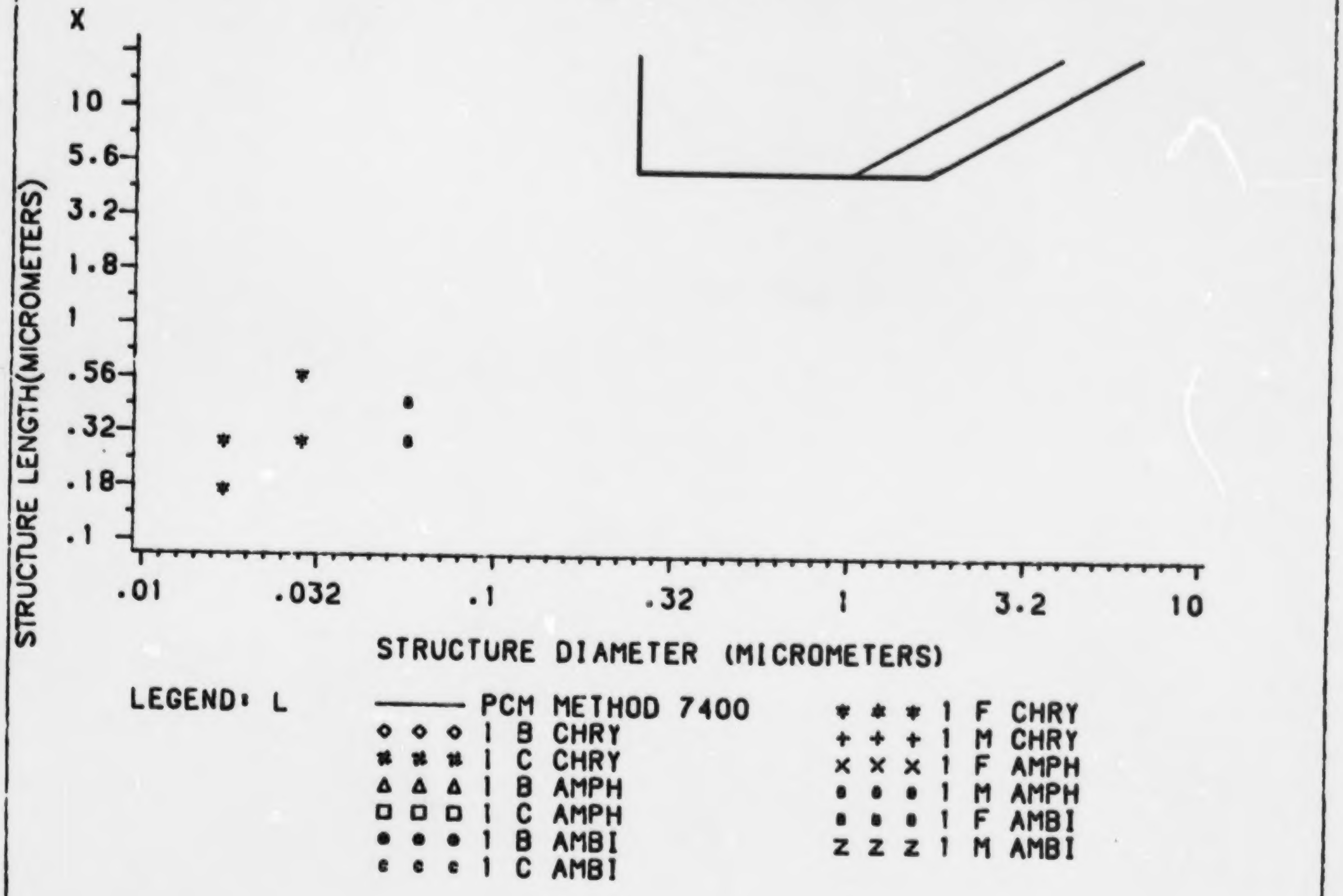
NIOSH: W1A - W3A & P1A - P3A

FILTER=W1A



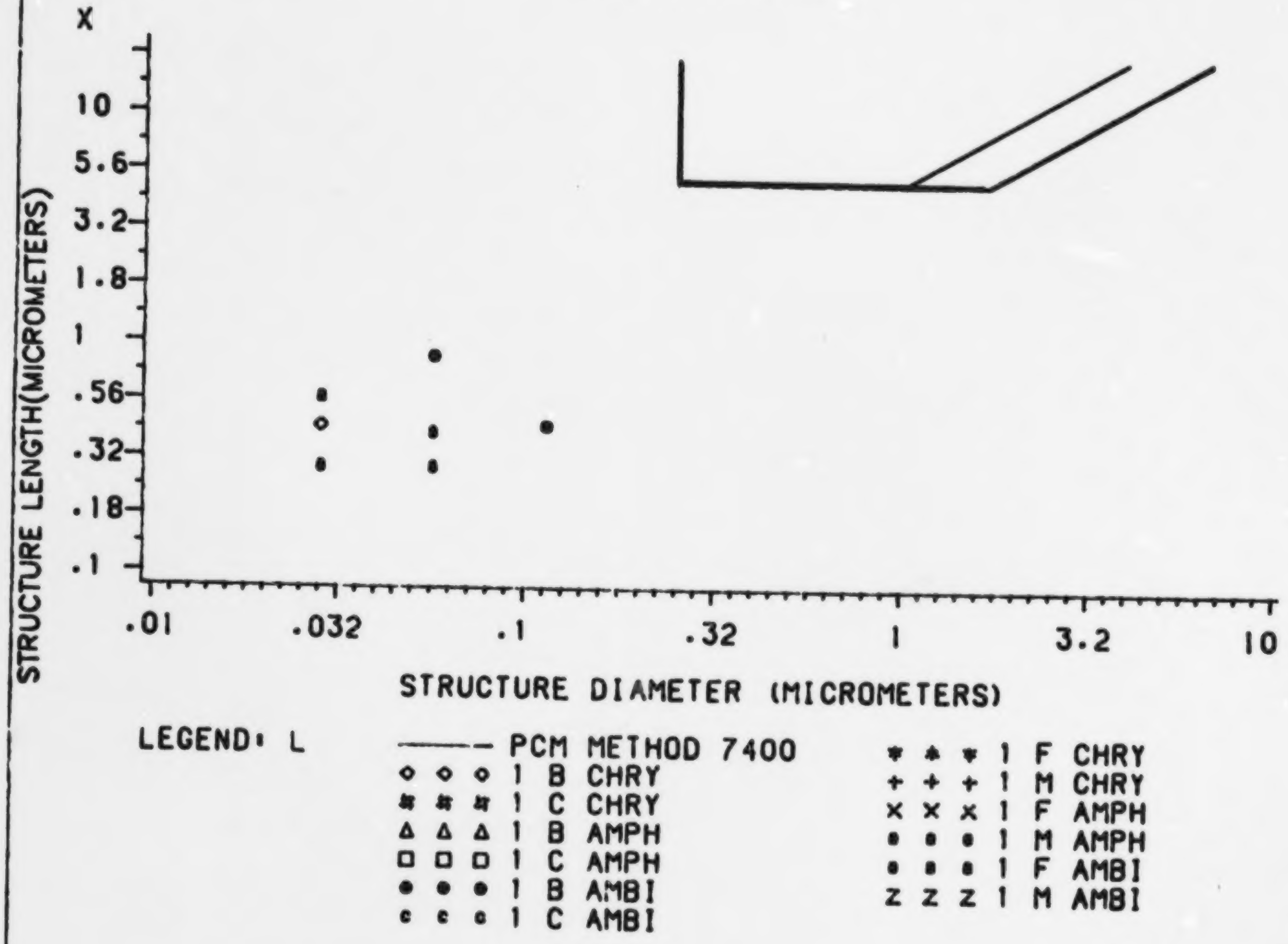
Distribution of structures in an Air Sample By TEM

DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=W1AN



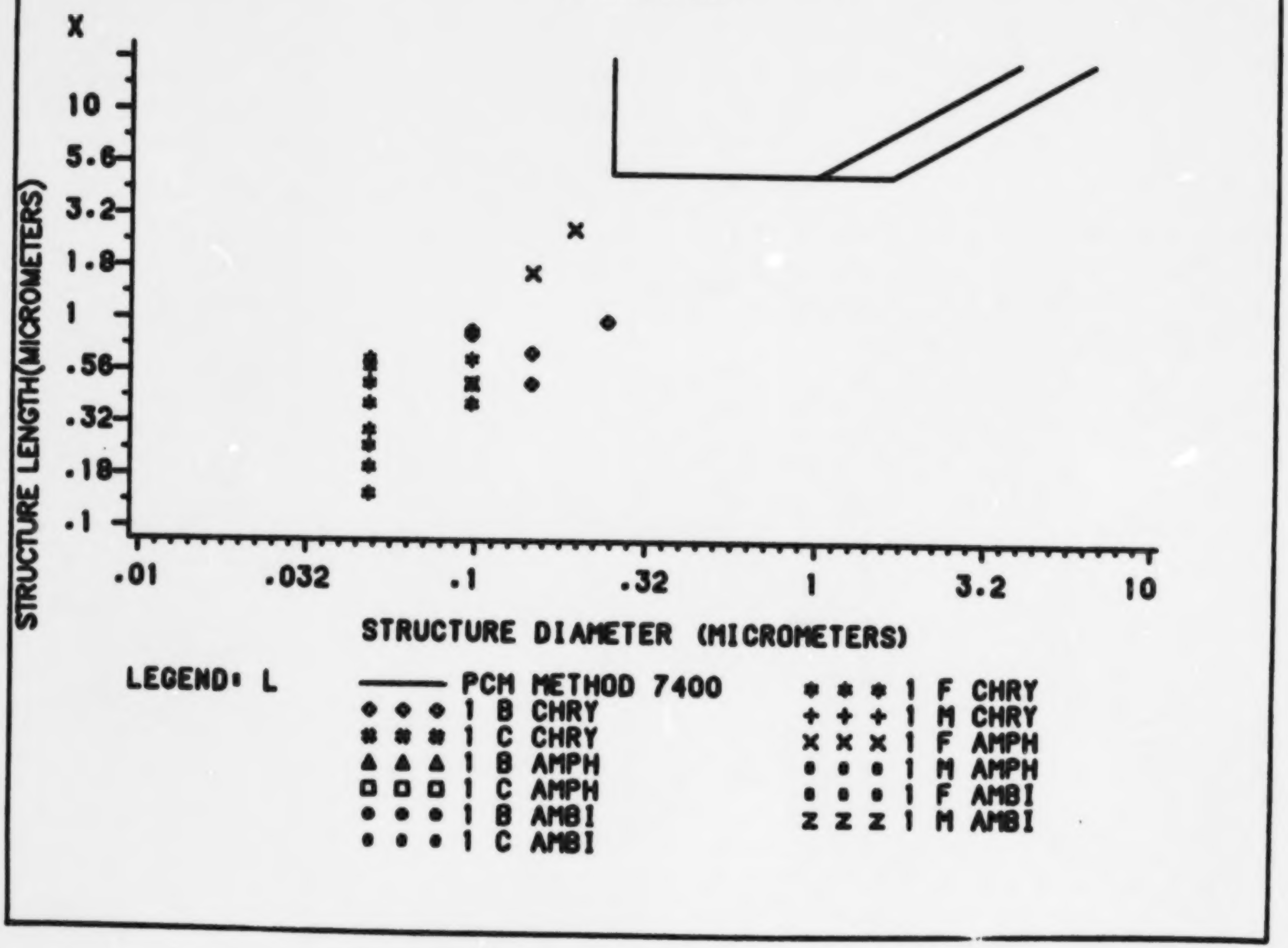
Distribution of structures in an Air Sample By TEM

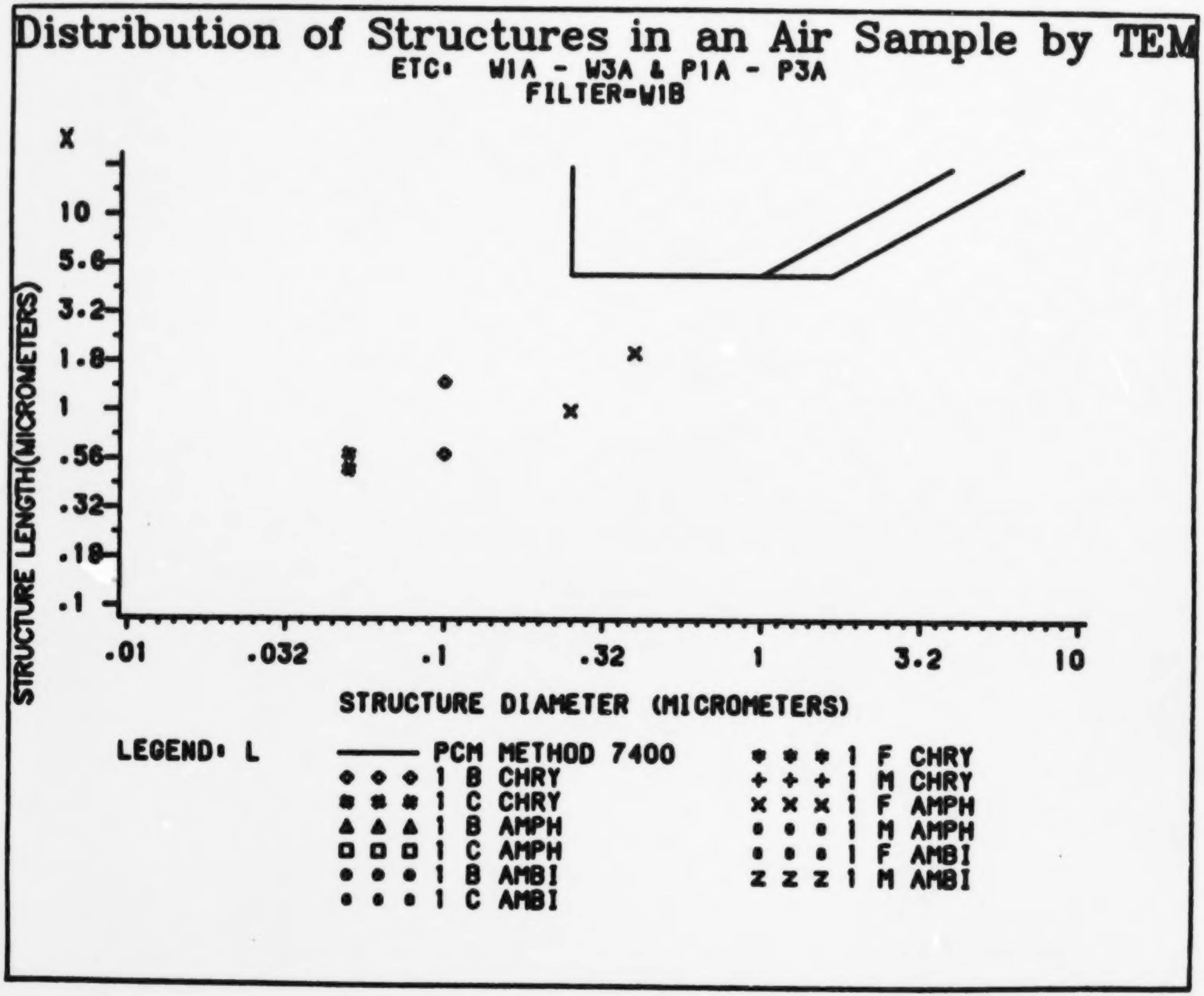
DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=W1AE



Distribution of Structures in an Air Sample by TEM

MCCRONE: W1B-W3B & P1B-P3B
FILTER=W1B

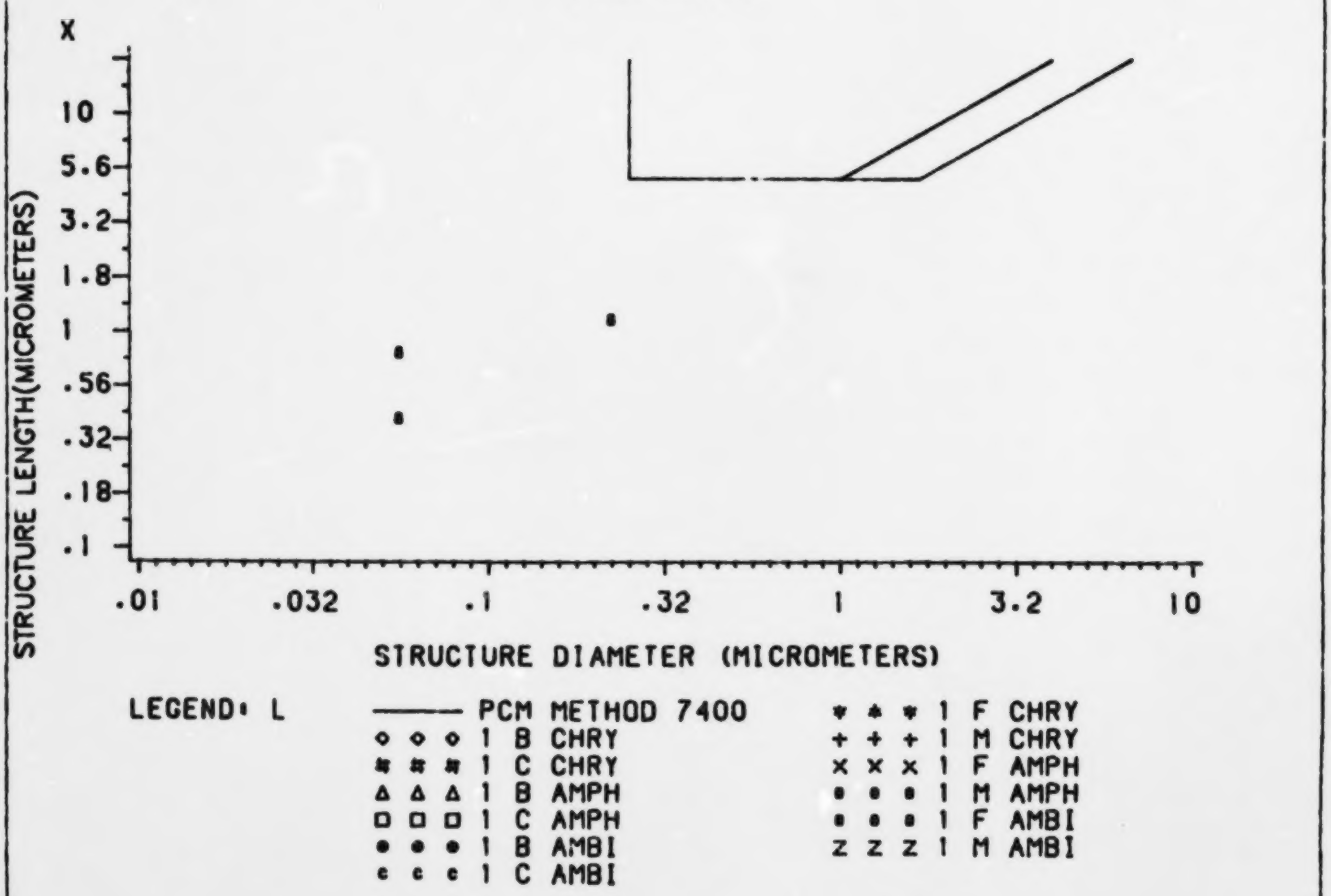



Distribution of Structures in an Air Sample by TEM
ETC: W1A - W3A & P1A - P3A
FILTER=W1B
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
STRUCTURE LENGTH(MICROMETERS)
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
—— PCM METHOD 7400
◆ ◆ ◆ 1 B CHRY
# 1 C CHRY
▲ ▲ ▲ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
● ● ● 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
● ● ● 1 M AMPH
● ● ● 1 F AMBI
Z Z Z 1 M AMBI

Distribution of Structures in an Air Sample by TEM

NIOSH: W1A - W3A & P1A - P3A

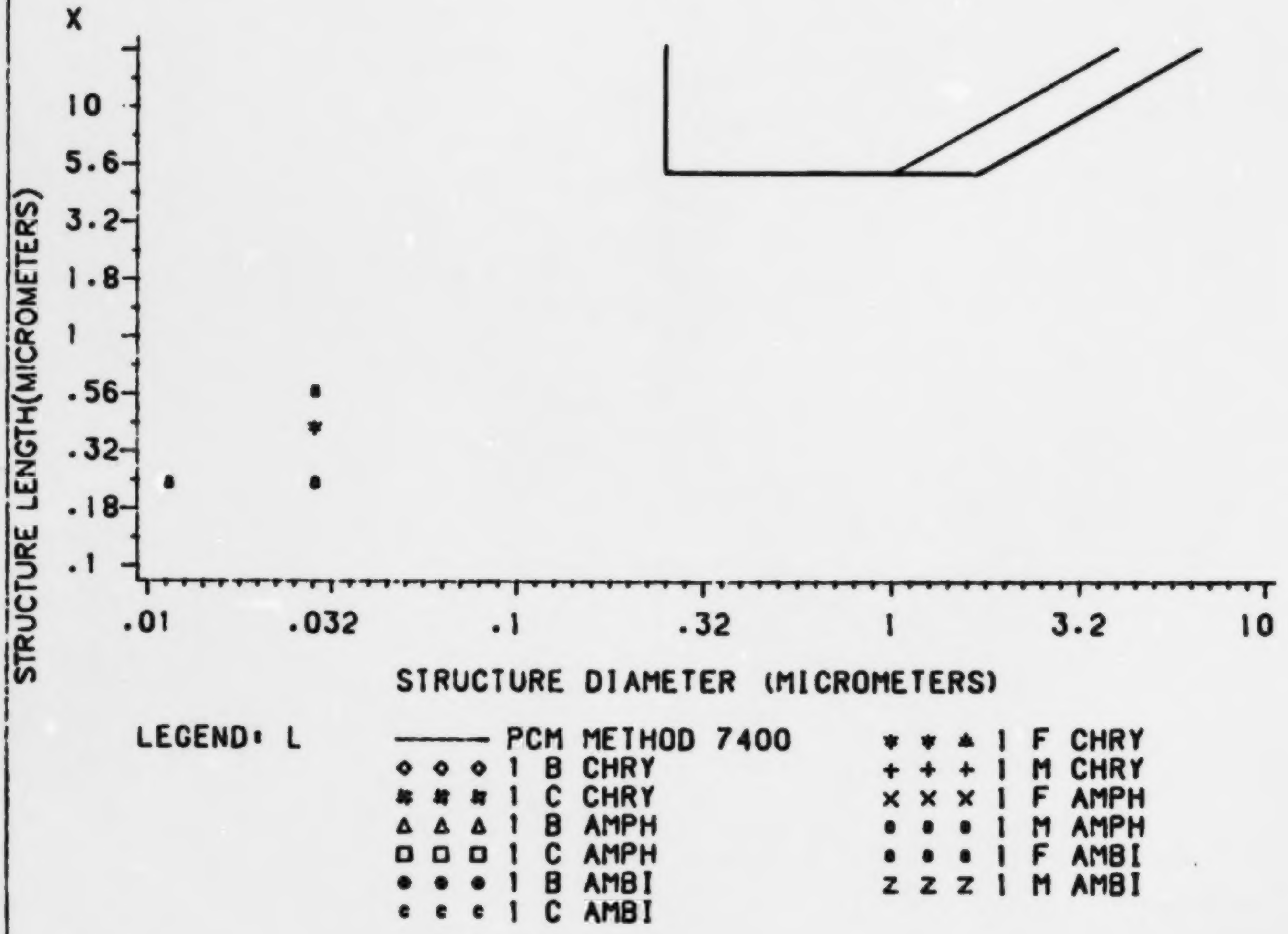
FILTER=W2A



Distribution of structures in an Air Sample By TEM

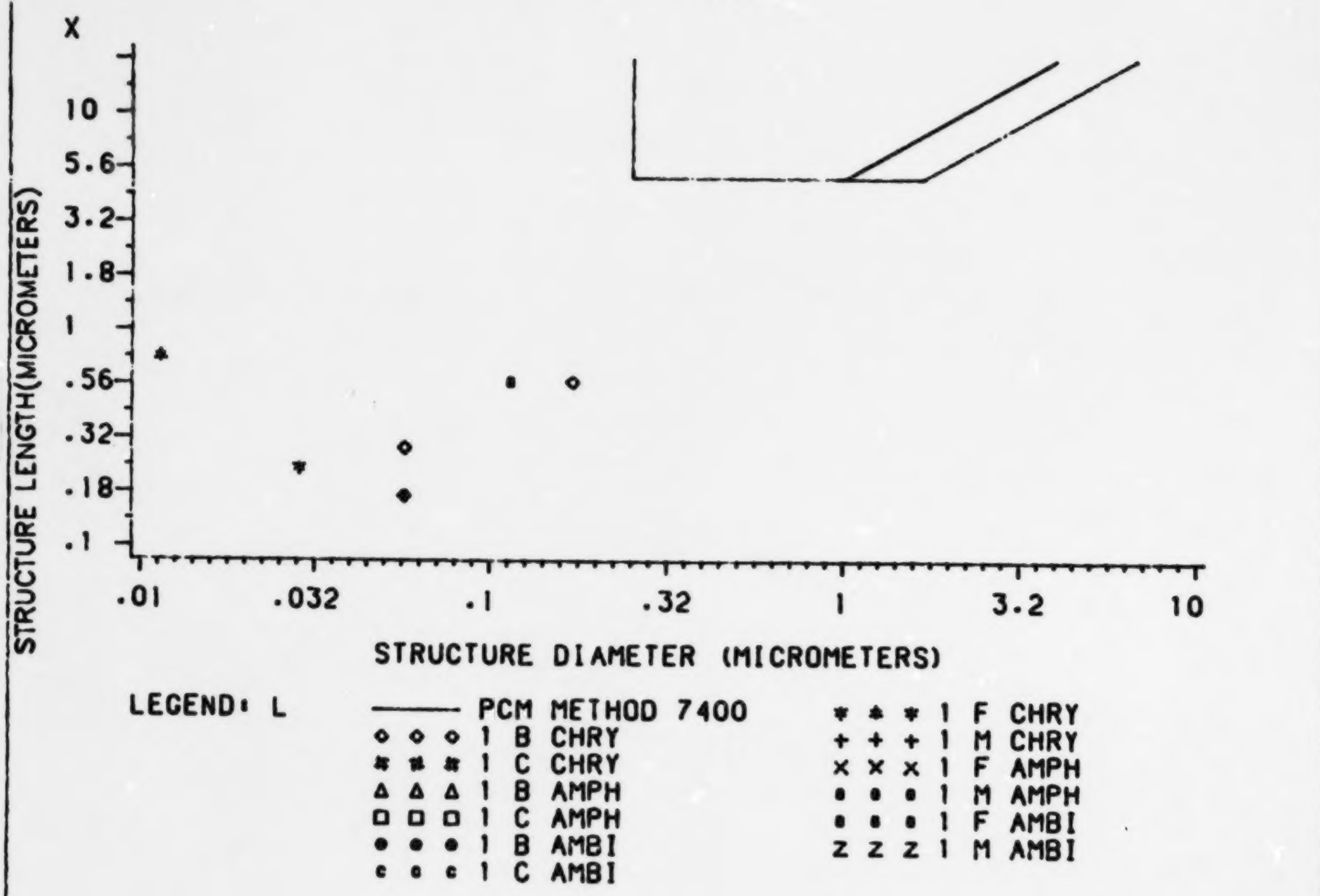
DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS

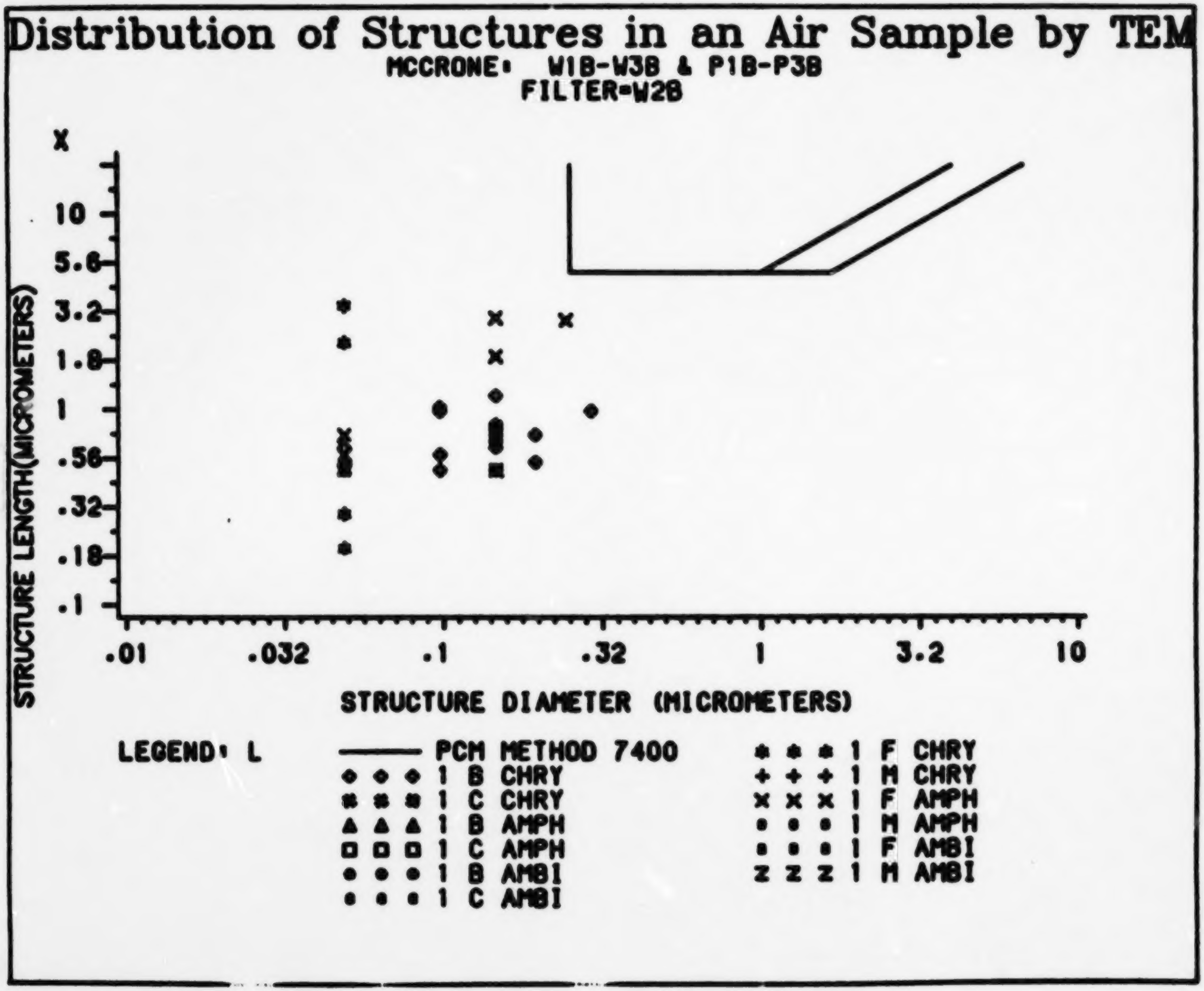
FILTER=W2AN



Distribution of structures in an Air Sample By TEM

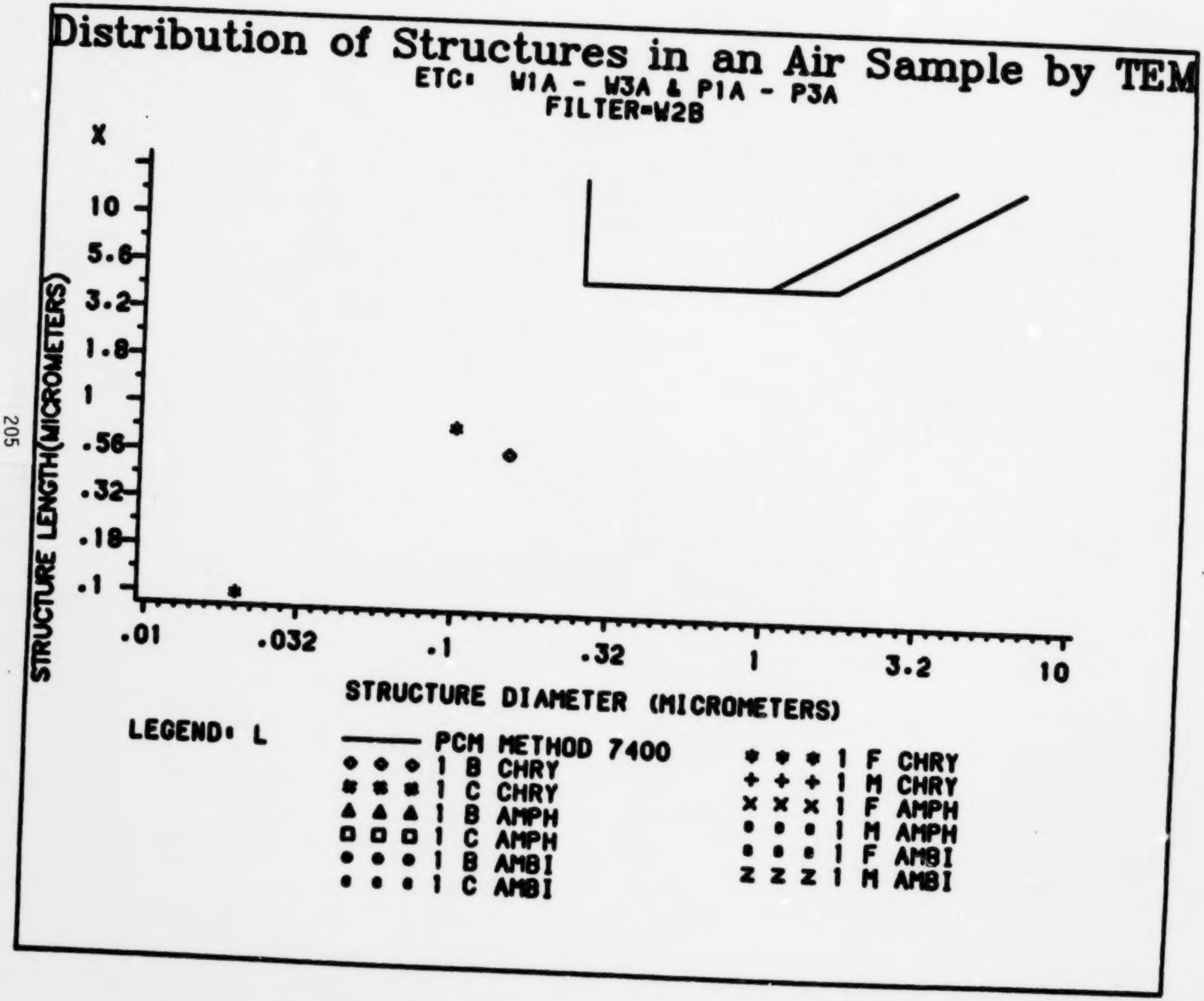
DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=W2AE





Distribution of Structures in an Air Sample by TEM

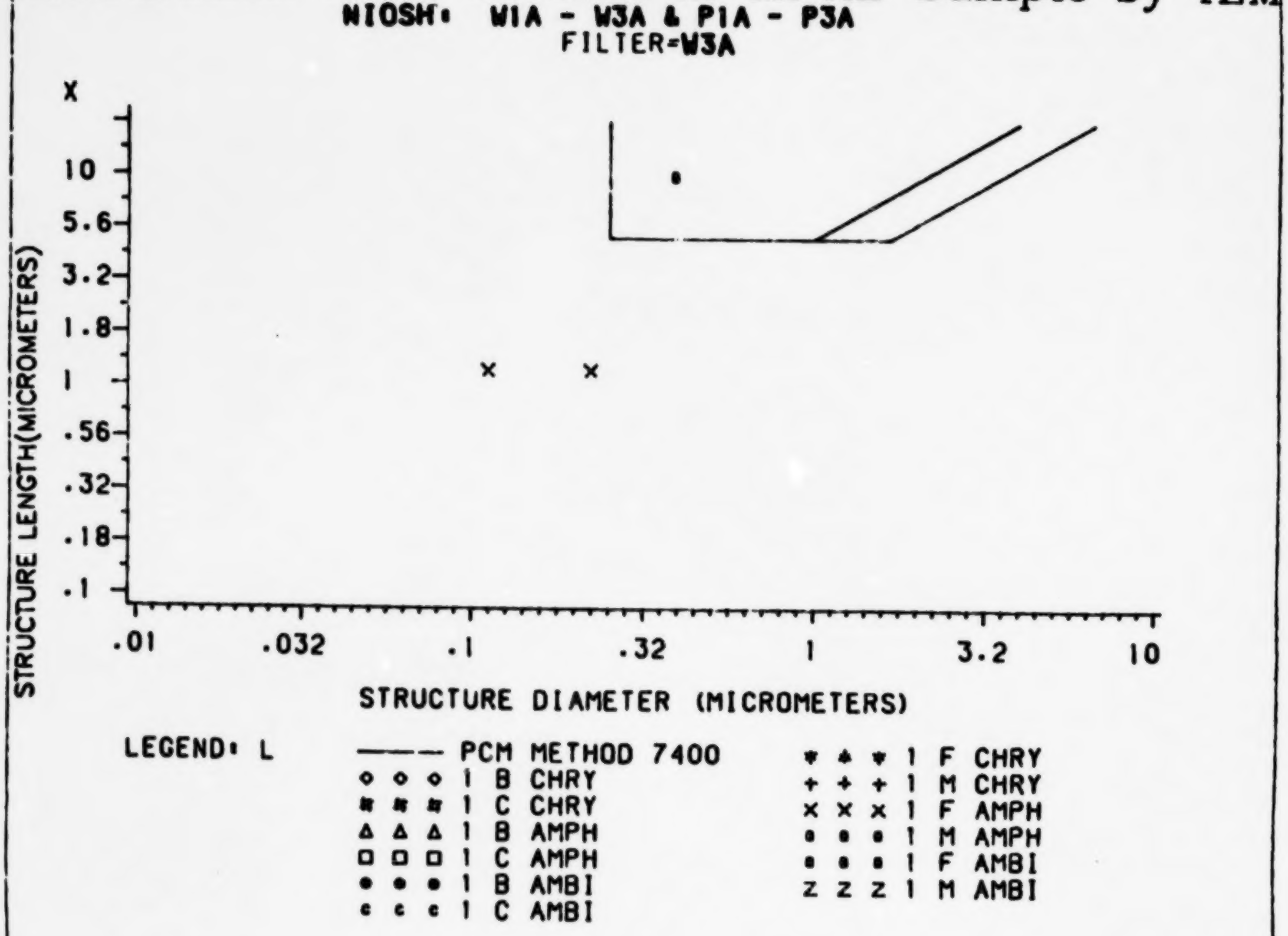
MCCRONE: W1B-W3B & P1B-P3B
FILTER=W2B

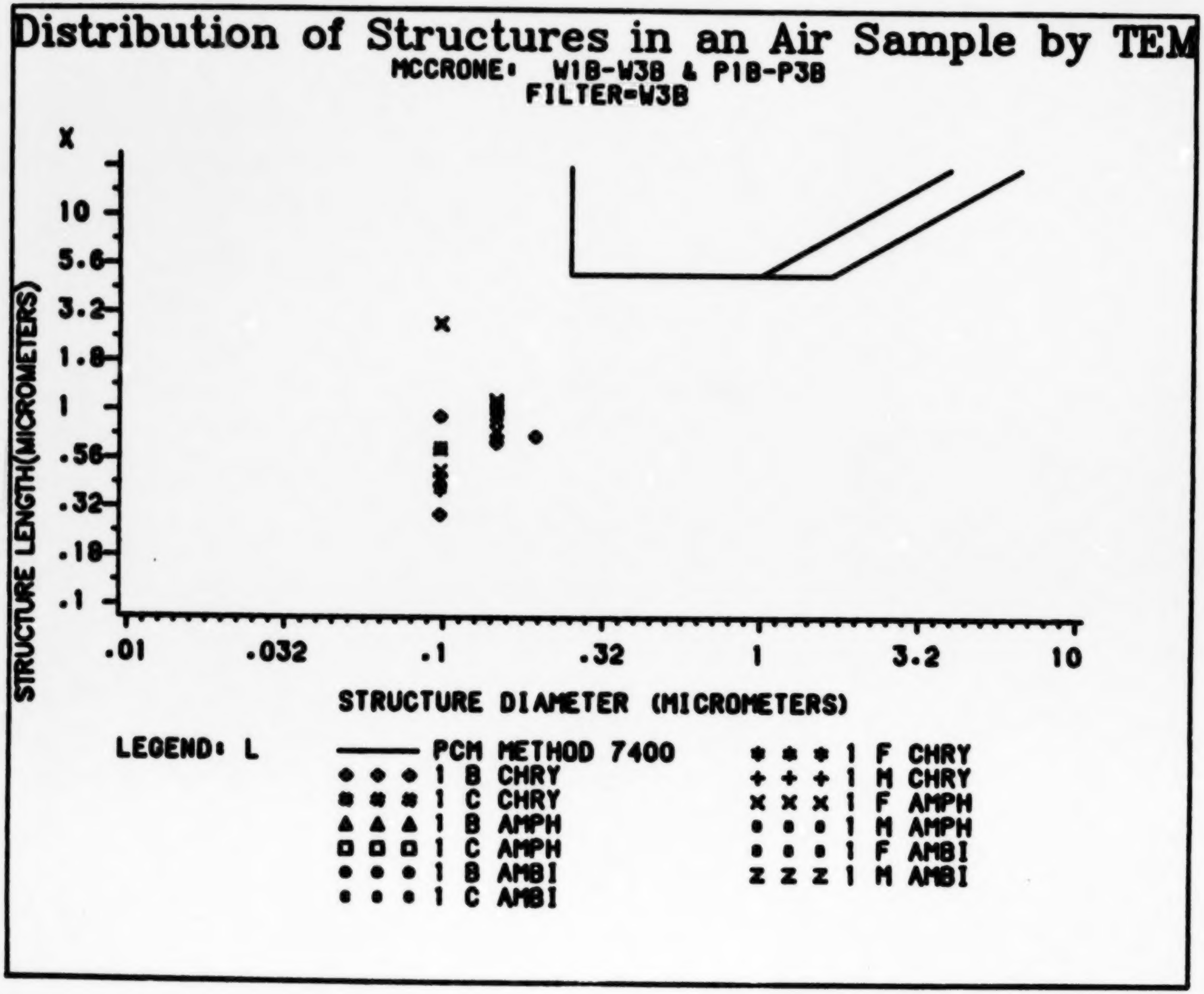


Distribution of Structures in an Air Sample by TEM

ETC: W1A - W3A & P1A - P3A
FILTER=W2B

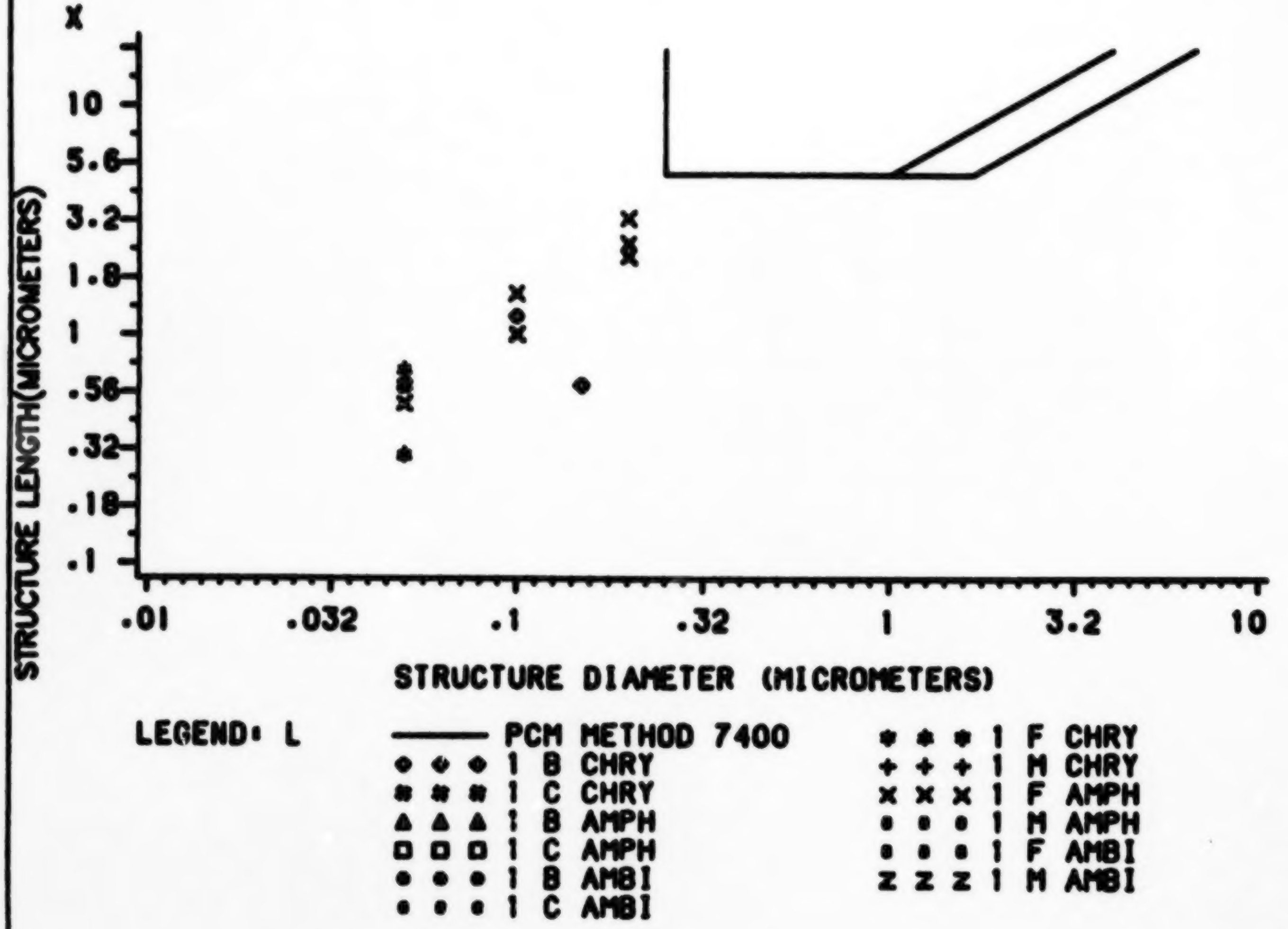
Distribution of Structures in an Air Sample by TEM




Distribution of Structures in an Air Sample by TEM
MCCRONE: W1B-W3B & P1B-P3B
FILTER=W3B
STRUCTURE LENGTH(MICROMETERS)
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
.01
.032
.1
.32
1
3.2
10
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
——— PCM METHOD 7400
◆ ◆ ◆ 1 B CHRY
# 1 C CHRY
▲ ▲ ▲ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
• • • 1 C AMBI
* * * 1 F CHRY
+ + + 1 M CHRY
x x x 1 F AMPH
• • • 1 M AMPH
• • • 1 F AMBI
Z Z Z 1 M AMBI

Distribution of Structures in an Air Sample by TEM

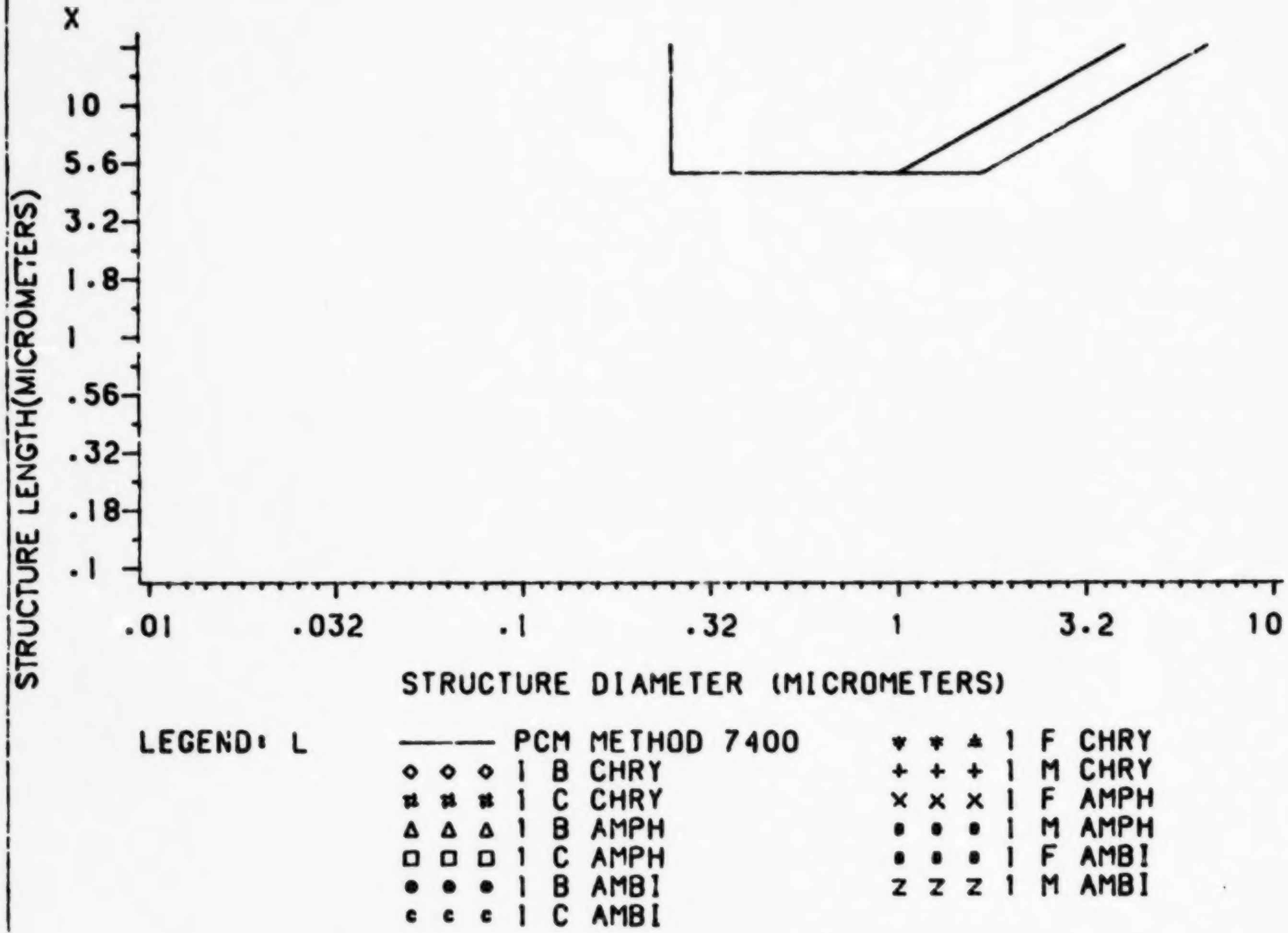
ETC: W1A - W3A & P1A - P3A
FILTER=W3B



Distribution of Structures in an Air Sample by TEM

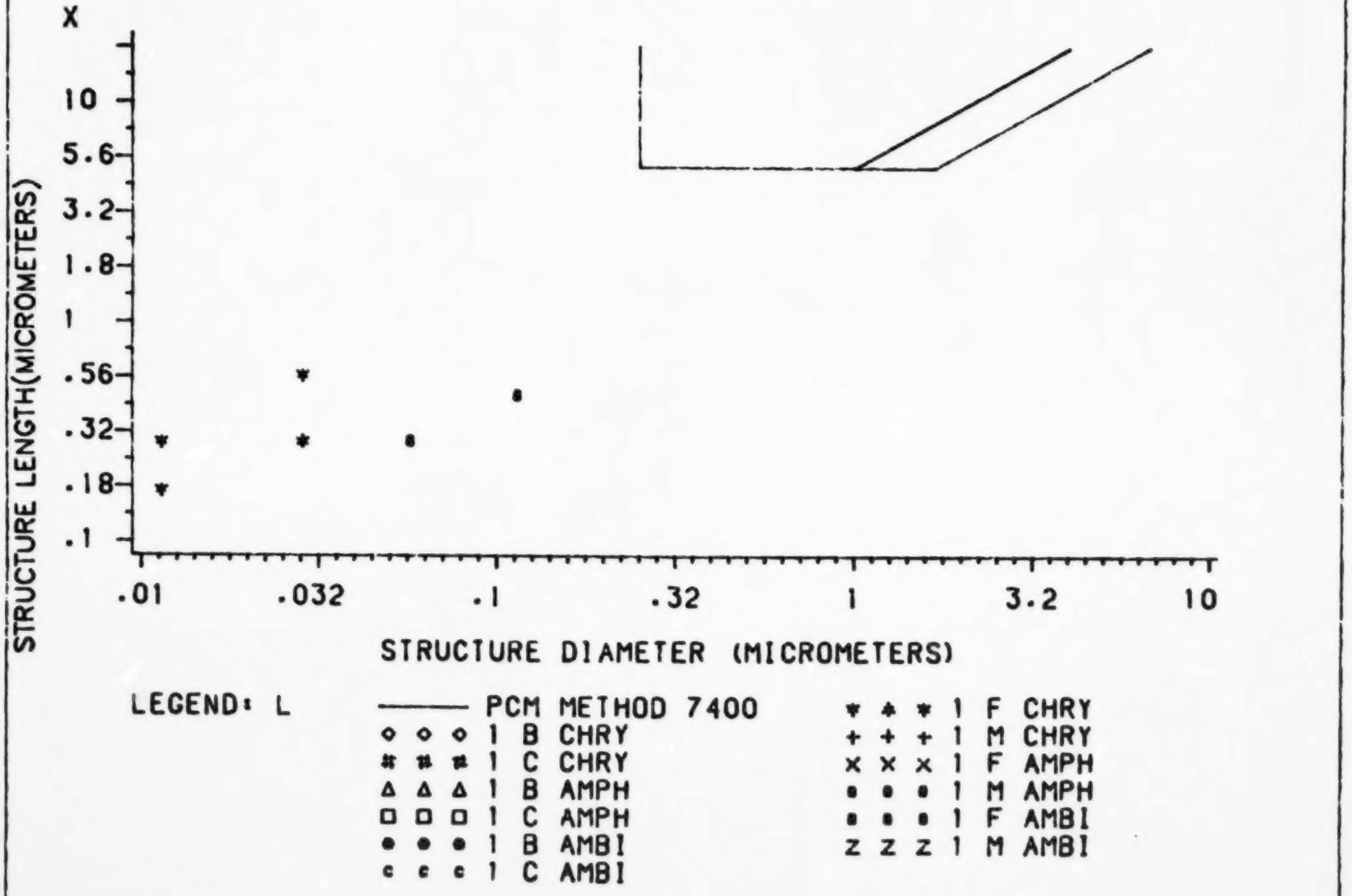
NIOSH: WIA - W3A & P1A - P3A

FILTER=**P1A**



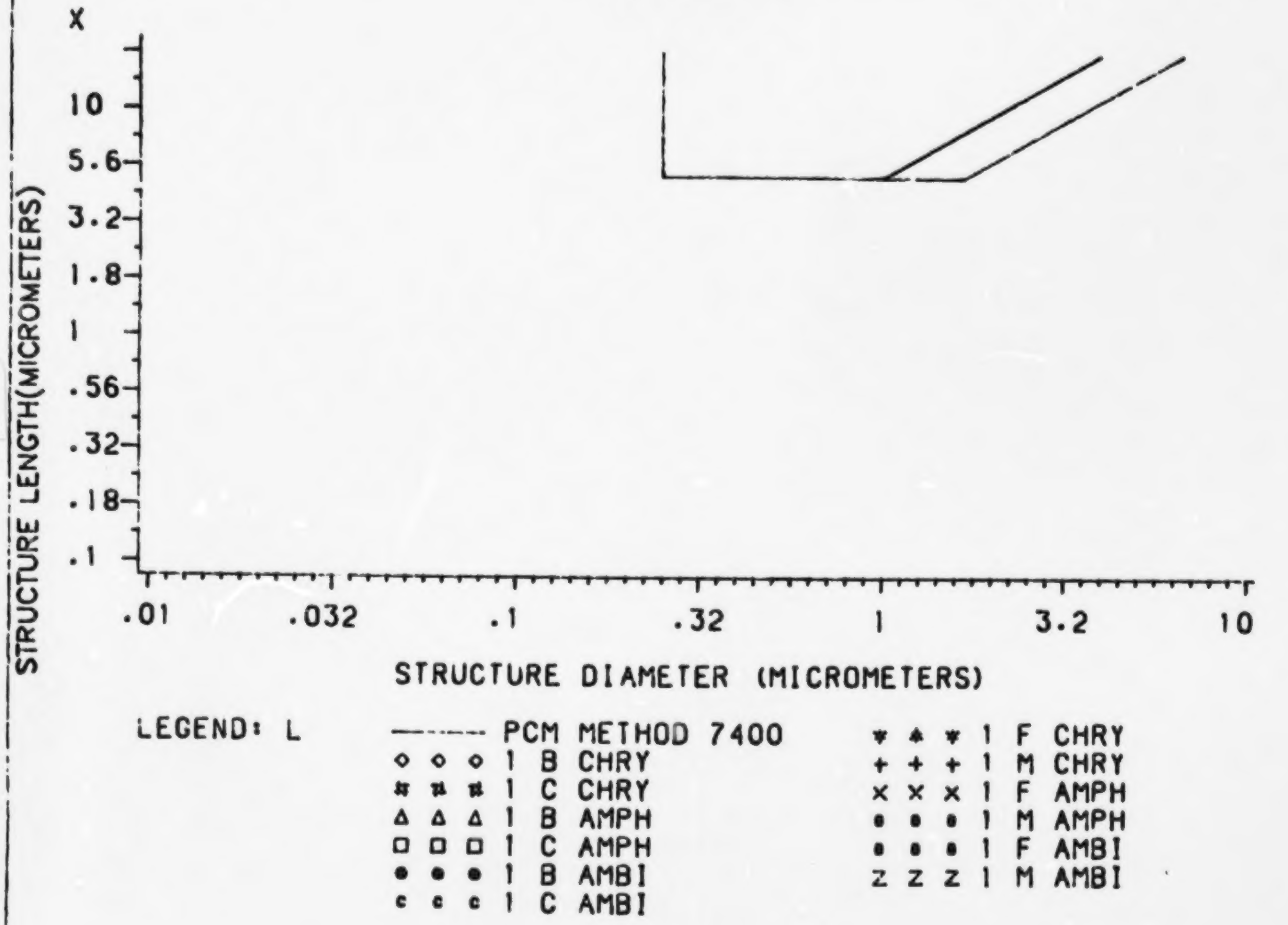
Distribution of structures in an Air Sample By TEM

DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=P1AN



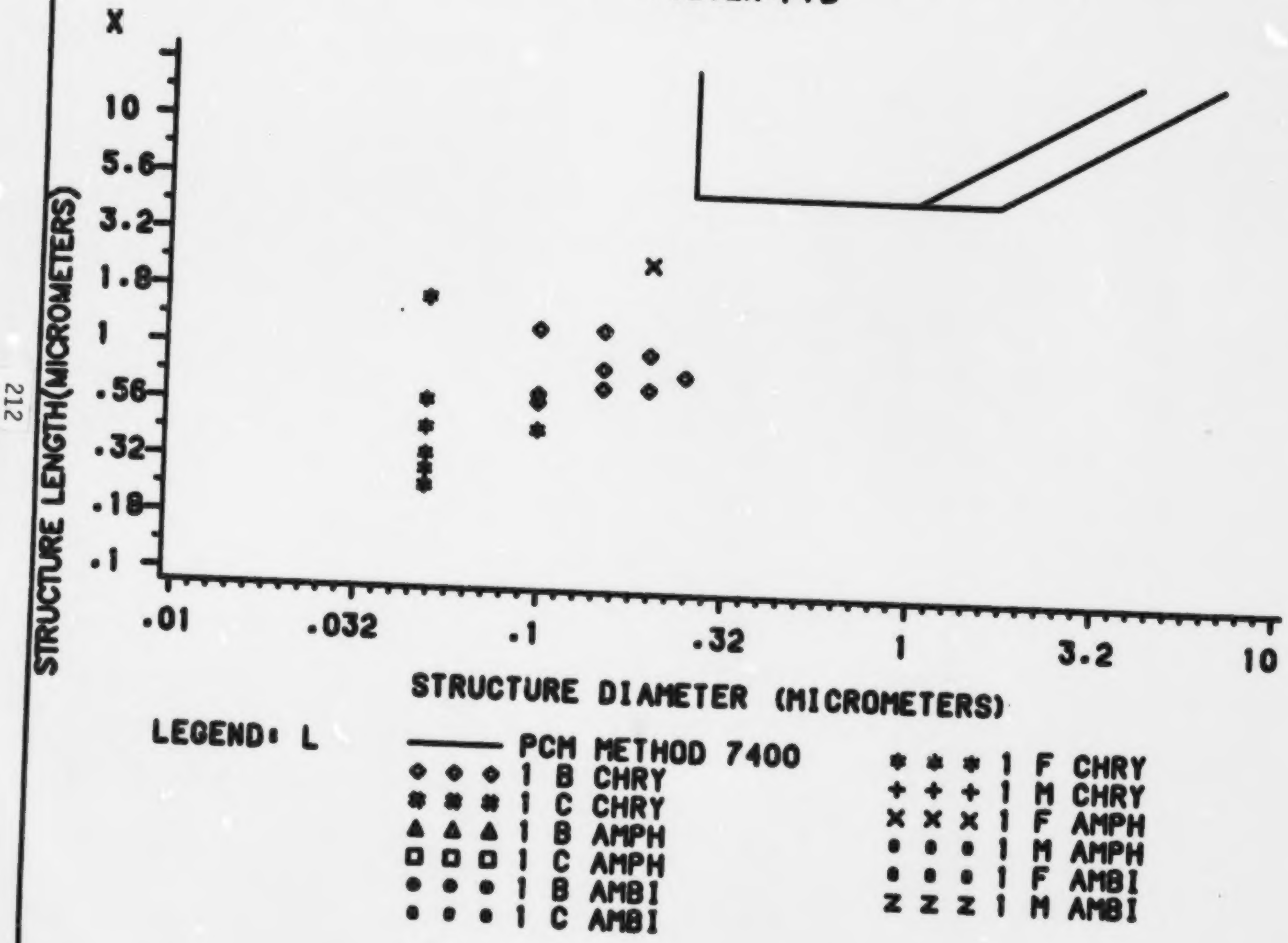
Distribution of structures in an Air Sample By TEM

DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=P1AE



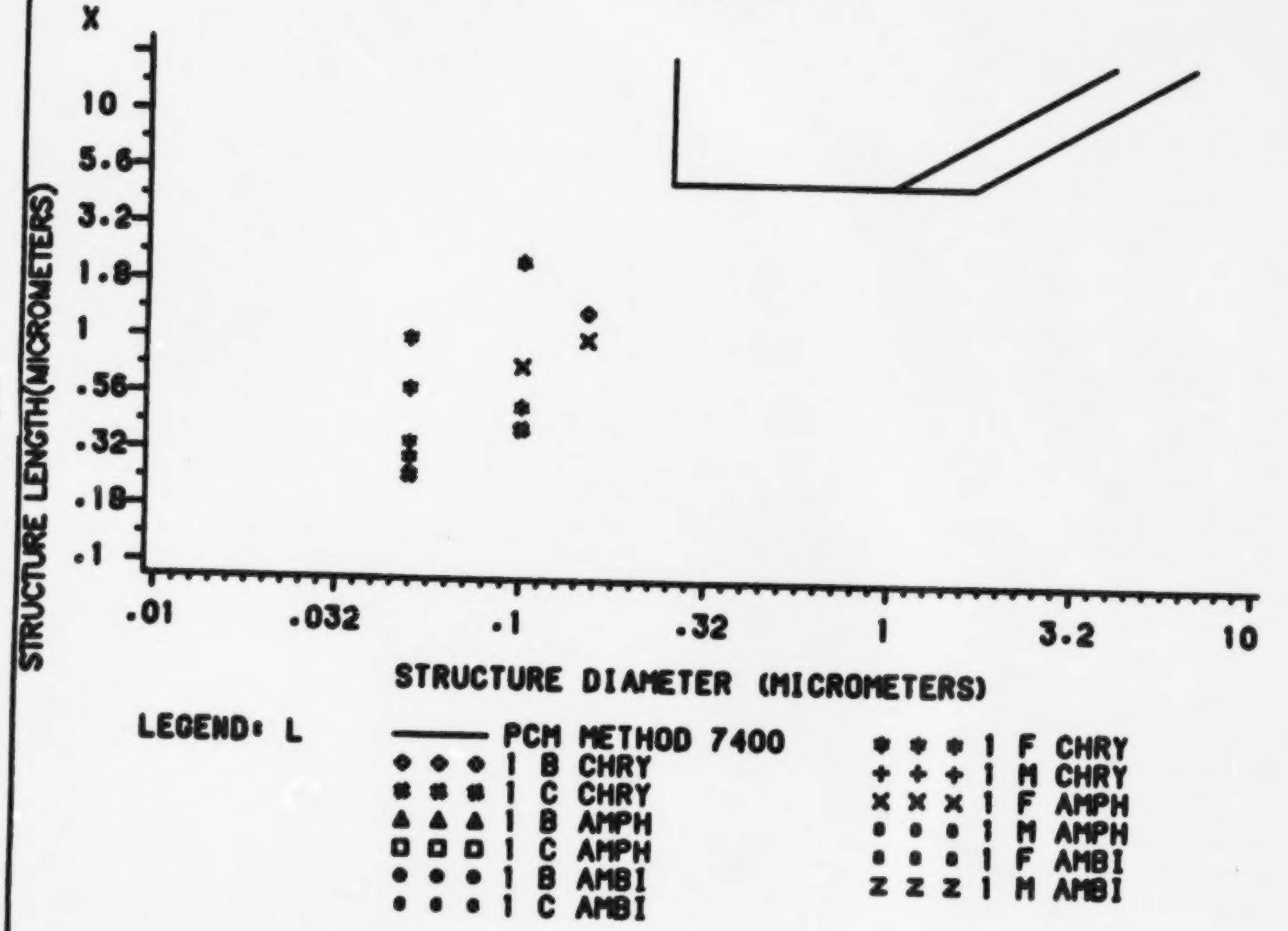
Distribution of Structures in an Air Sample by TEM

MCCRONE: W1B-W3B & P1B-P3B
FILTER=P1B



Distribution of Structures in an Air Sample by TEM

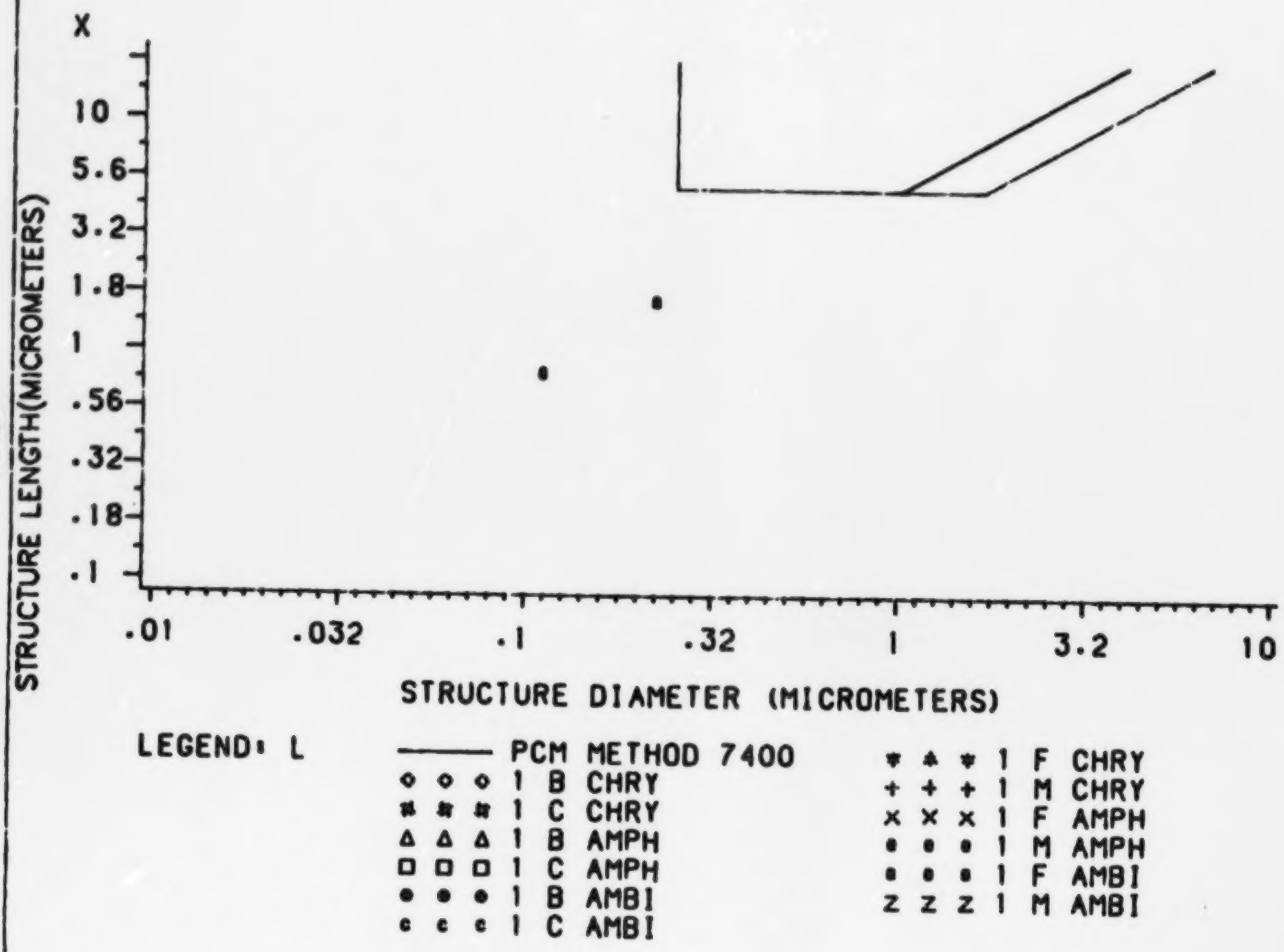
ETC: W1A - W3A & P1A - P3A
FILTER=P1B



Distribution of Structures in an Air Sample by TEM

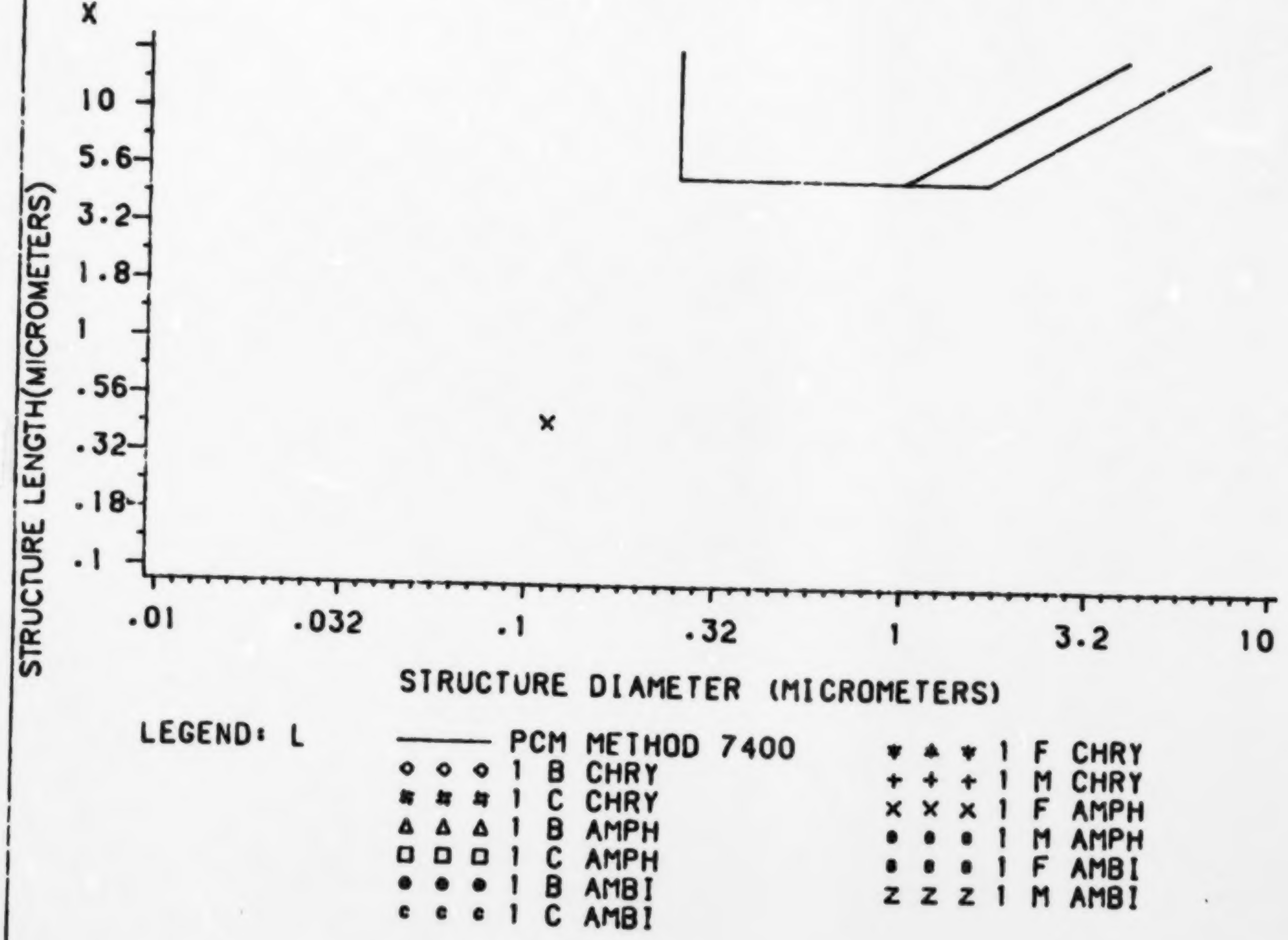
NIOSH: WIA - W3A & PIA - P3A

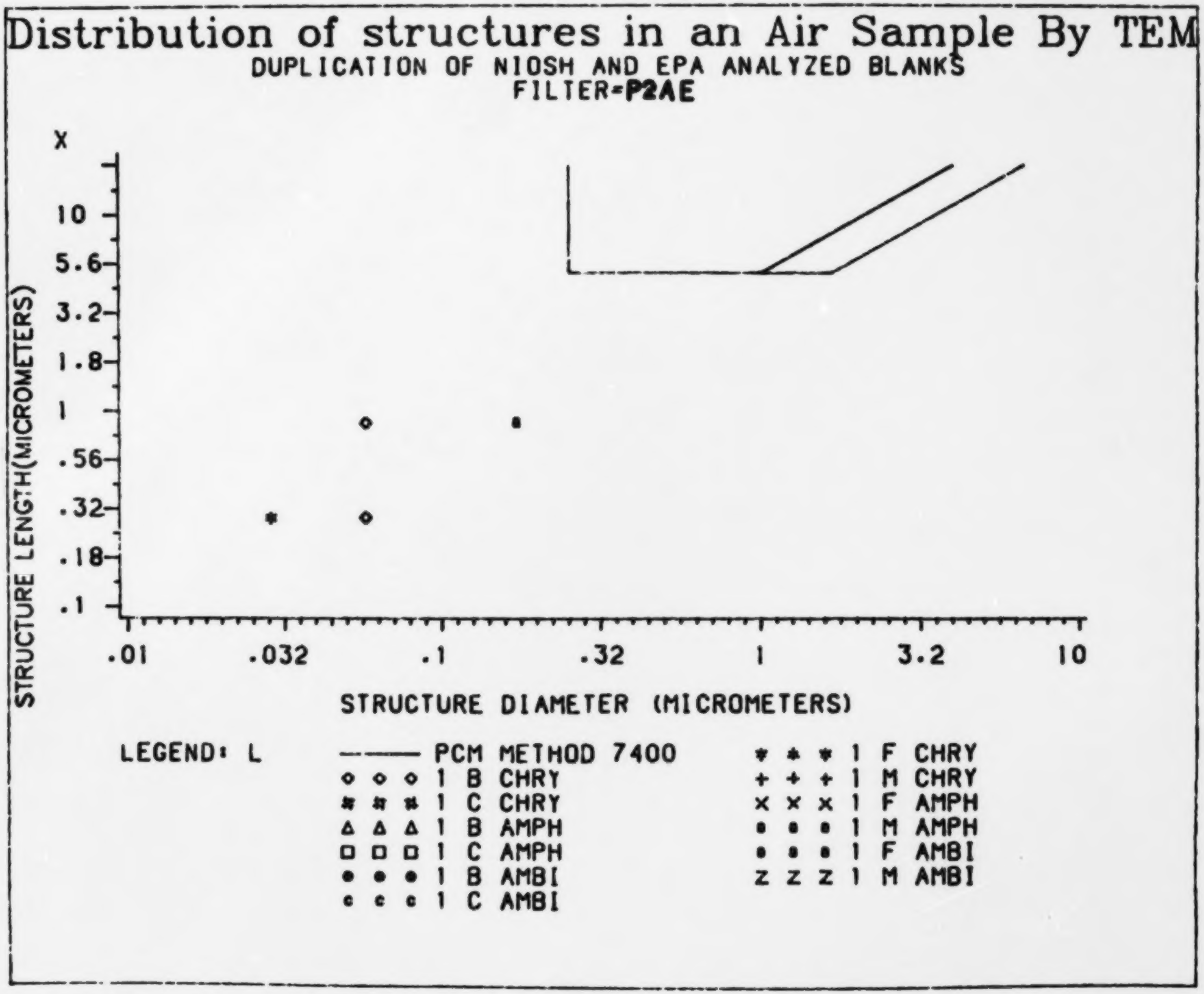
FILTER=**P2A**



Distribution of structures in an Air Sample By TEM

DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=P2AN



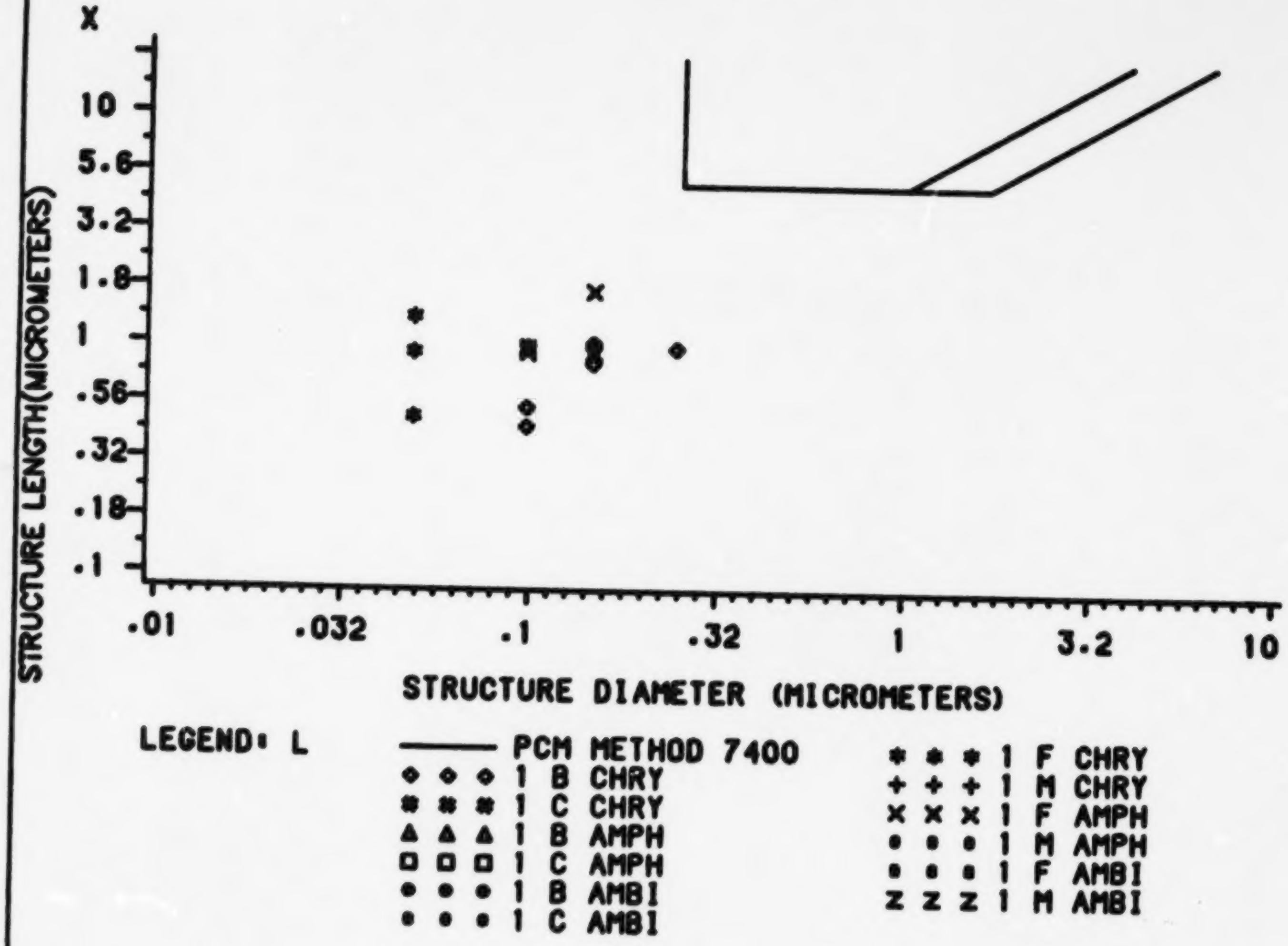


Distribution of structures in an Air Sample By TEM

DUPLICATION OF NIOSH AND EPA ANALYZED BLANKS
FILTER=P2AE

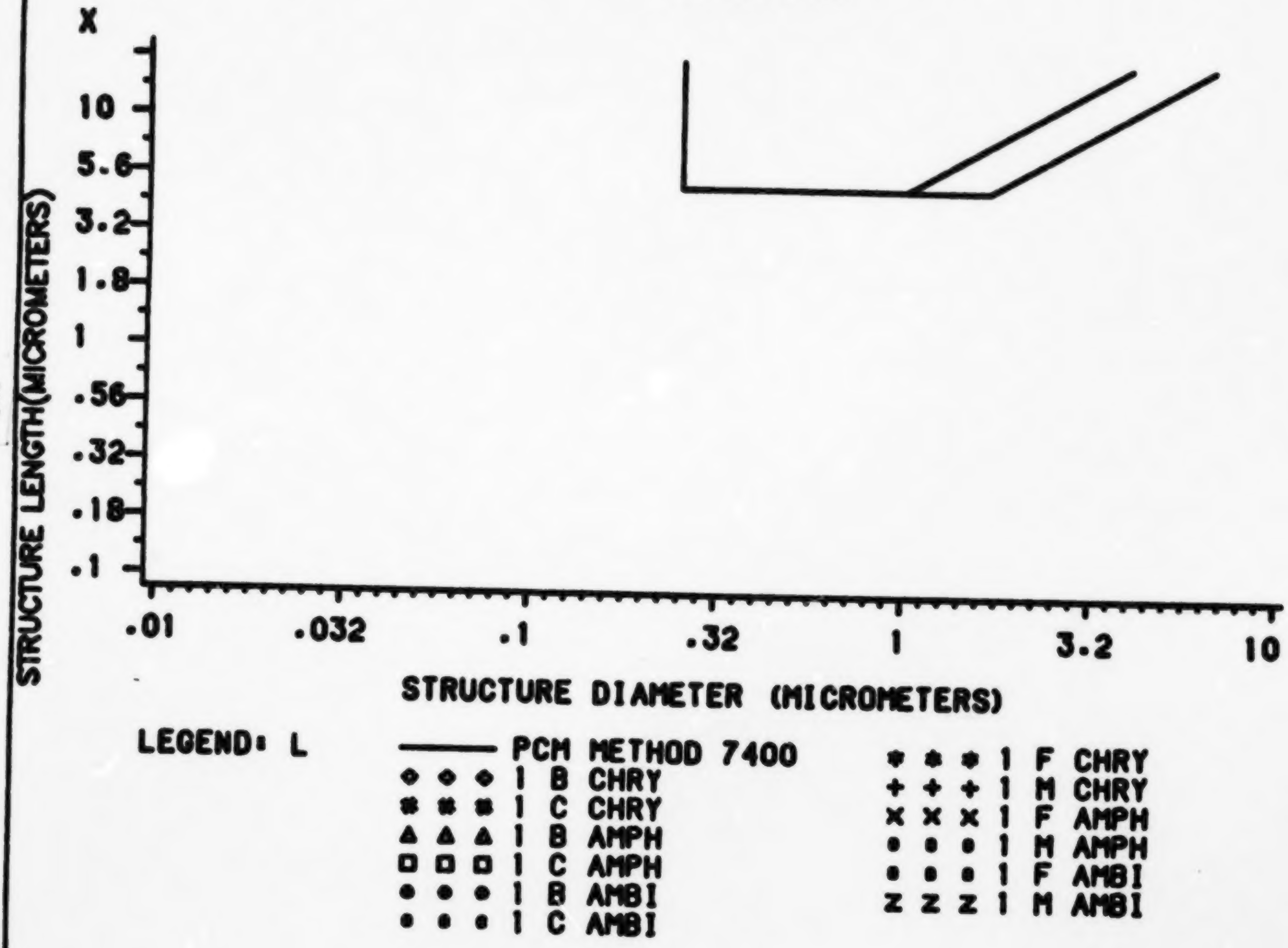
Distribution of Structures in an Air Sample by TEM

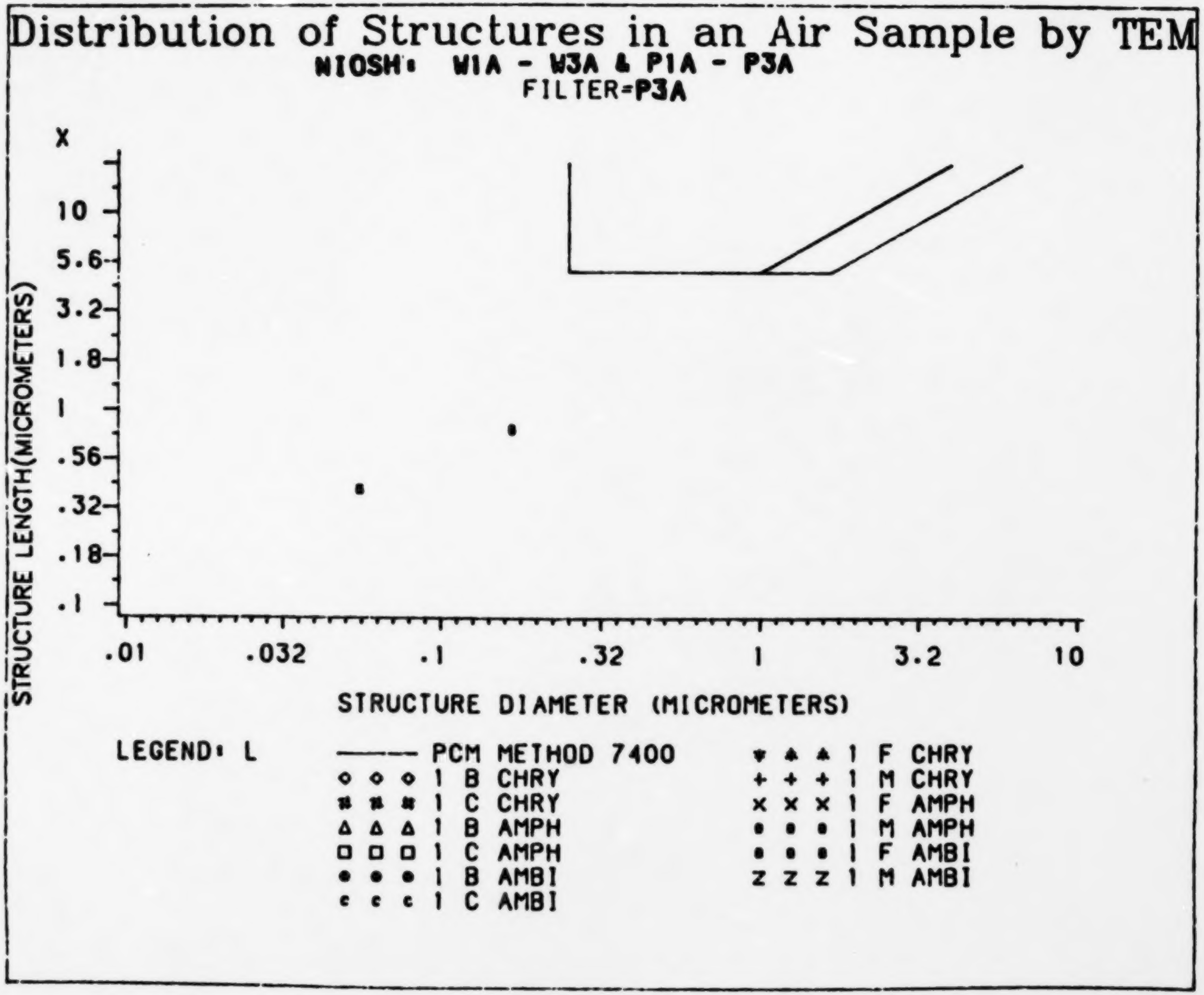
MCCRONE: W1B-W3B & P1B-P3B
FILTER=P2B



Distribution of Structures in an Air Sample by TEM

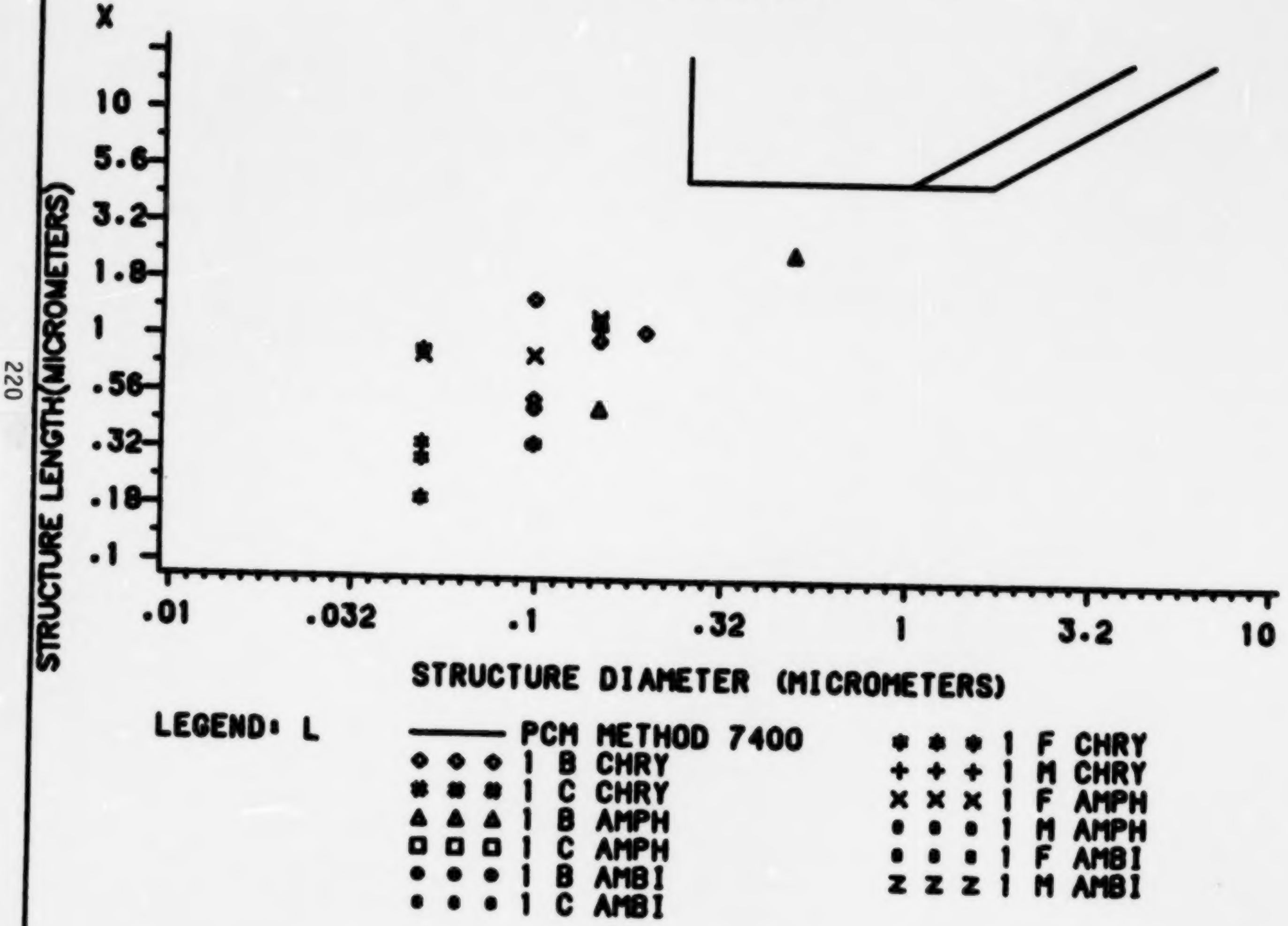
ETC: W1A - W3A & P1A - P3A
FILTER=P2B




Distribution of Structures in an Air Sample by TEM
NIOSH: W1A - W3A & P1A - P3A
FILTER=P3A
X
10
5.6
3.2
1.8
1
.56
.32
.18
.1
.01
.032
.1
.32
1
3.2
10
STRUCTURE LENGTH(MICROMETERS)
STRUCTURE DIAMETER (MICROMETERS)
LEGEND: L
——— PCM METHOD 7400
◇ ◇ ◇ 1 B CHRY
⌗ ⌗ ⌗ 1 C CHRY
△ △ △ 1 B AMPH
□ □ □ 1 C AMPH
● ● ● 1 B AMBI
c c c 1 C AMBI
▲ ▲ ▲ 1 F CHRY
+ + + 1 M CHRY
× × × 1 F AMPH
• • • 1 M AMPH
▪ ▪ ▪ 1 F AMBI
z z z 1 M AMBI

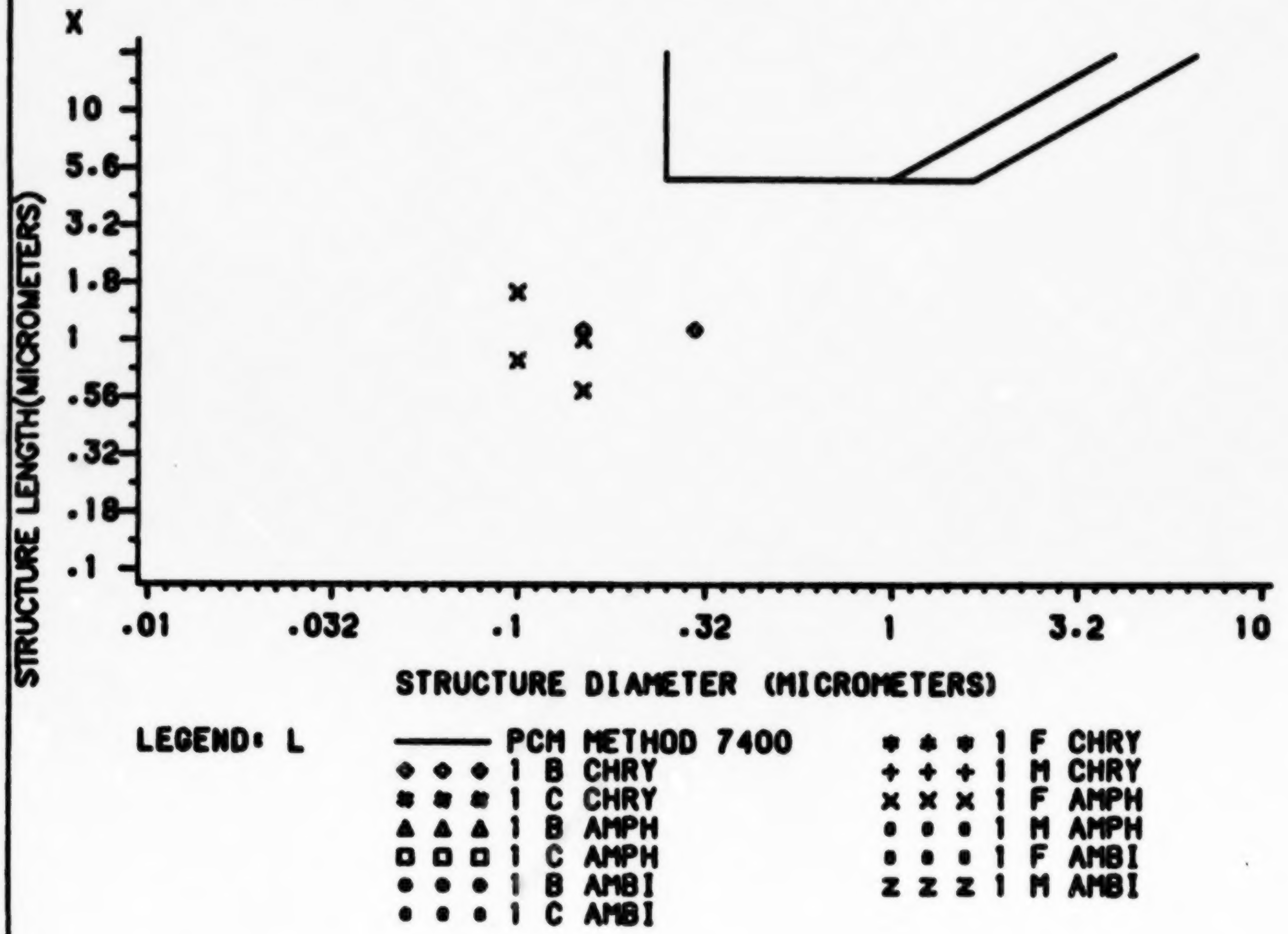
Distribution of Structures in an Air Sample by TEM

MCCRONE: W1B-W3B & P1B-P3B
FILTER=P3B



Distribution of Structures in an Air Sample by TEM

ETC: W1A - W3A & P1A - P3A
FILTER=P3B



APPENDIX K

ISSUES TO BE RESOLVED FOLLOWING PEER REVIEW CONFERENCE ON SAMPLE CONTAMINATION DURING AIRBORNE ASBESTOS SAMPLING

Michael E. Beard
Environmental Monitoring Systems Laboratory
Research Triangle Park, North Carolina 27711

Several key issues related to sampling and analysis of airborne asbestos have been raised by the discussions at this conference. These issues are identified and possible approaches to their resolution are briefly discussed below.

Contamination of Blanks

At least three types of blank contamination have been identified: (1) contaminated filter media as obtained from the manufacturer, (2) contaminated filters due to exposure to airborne asbestos at the sampling site, and (3) contaminated filters in the laboratory during sample preparation. These sources of contamination can be avoided by careful screening of all filter media, materials and reagents used in the sampling and sample preparation. This screening must be conducted prior to any sampling efforts. Procedures for sampling and analysis should identify critical areas where contamination is possible and should prescribe acceptance testing to identify and avoid such contamination. EPA, NBS, NIOSH and other agencies should encourage the development and marketing of "asbestos free" filter media for asbestos monitoring.

Electrostatic Charge Effects on Samples

The effects of electrostatic charges on sampling may be the most critical variable discussed here. Evidence presented at this meeting and at others have suggested that asbestos may be attracted to filter media or to the filter cassettes by electrostatic charge. This effect may result in either greatly increased or reduced asbestos fibers on the filter during analysis. Efforts to understand and overcome these effects must be undertaken. The use of antistatic materials or devices in overcoming this problem must be investigated and clearly understood before than can be effectively controlled.

Analytical Protocol

The 1984 Yamate TEM procedure prescribes counting 10 grid openings at 20,000X. Some analysts (here and elsewhere) are using various magnifications and counting areas for improving counting statistics, for reducing analysis time and operator fatigue, and for various other purposes. While each analyst may feel comfortable with his own modifications, his fellow analysts may not share his enthusiasm. The accuracy of the analysis and the convenience of the counting protocol to the analyst must be carefully balanced to insure the integrity of the data. A performance standard should be incorporated into the Yamate procedure to demonstrate that variations in the analytical protocol are acceptable and capable of producing consistent results.

Statistical variability in the analytical data needs to be clearly defined. A data reporting scheme similar to that used by Dr. Richard J. Lee of ETC, Inc. should be considered and included in the reporting of all asbestos monitoring data (see attachment).

Filter Media

The Yamate procedure prescribes the use of polycarbonate filters (Nuclepore) for sample collection and the direct transfer technique to the grid in order to preserve the integrity of the sample. A direct transfer technique from cellulose ester filters (Millipore, Nuclepore, Gelman, etc.) described by Burdett & Rood is gaining in popularity among several investigators. This procedure offers several benefits including (1) ease of handling during sampling, (2) more rapid sample preparation, and (3) possibly reduced incidences of filter contamination. This procedure is promising but its relationship to samples collected on polycarbonate needs to be defined and understood before a decision to adopt this modification is made.

Epilogue

Resolution of the problems outlined here requires carefully planned research. Such research should be planned, conducted, and reviewed by a panel of experts, such as those attending this conference. Research dollars being at a premium, EPA and other agencies should cooperate in these studies to maximize the effort.

ATTACHMENT

NONDETECTABLE SAMPLE: Fewer than five asbestos fibers detected. Assuming a blank count corresponding to one particle and Poisson statistics (a= 0.5), this implies that the result is indistinguishable from background.

INDETERMINATE SAMPLE: Between 5 and 11 asbestos fibers detected. In this range, the count is distinguishable from background but the probability that a repetition of the analysis could yield a result indistinguishable from background is greater than 5 percent.

DETECTABLE SAMPLE: More than 11 asbestos fibers detected. The probability that a repetition of the analysis would be indistinguishable from background is less than 5 percent.

MEASURABLE SAMPLE: More than 100 asbestos fibers detected. This specifies that uncertainty that a repetition of the analysis would be indistinguishable from background is less than 10 percent.

If an air sample contained an indeterminate, a detectable, or a measurement amount of asbestos, an air concentration was calculated based on the amount of asbestos detected on the air filter and the volume of air sampled. Otherwise, the air sample was designated as containing a nondetectable amount of asbestos.

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